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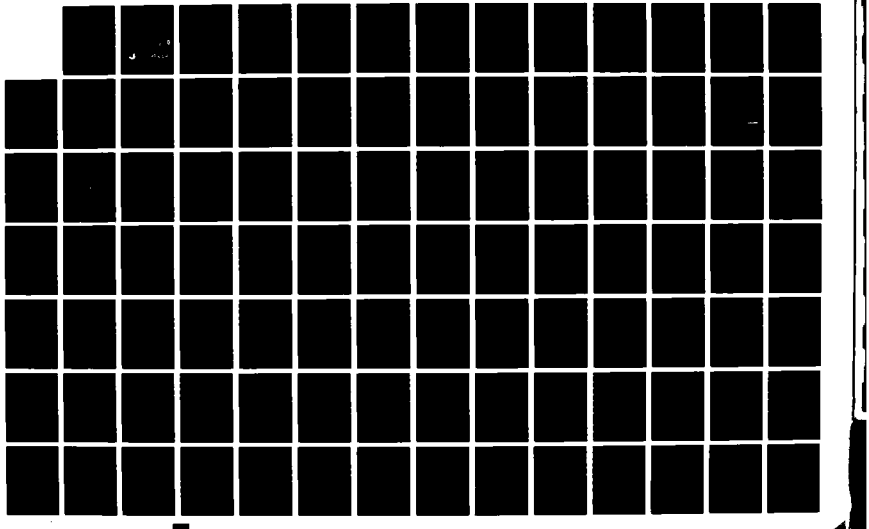

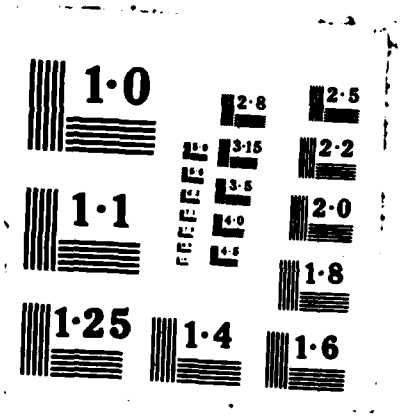
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# DEGRADATION OF JET AND MISSILE FUELS BY AQUATIC MICROBIAL COMMUNITIES

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See page ii

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## EXECUTIVE SUMMARY

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Keywords - water pollution; environmental impact





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## PREFACE

This research was conducted at the United States Environmental Protection Agency, Environmental Research Laboratory, Gulf Breeze, Florida, under Interagency Agreement AR-57-F-2-A-016 with the United States Air Force.

This report covers work performed between October 1982 and October 1984. The AFESC/RDVC Project Officer was Lt. Robert Beggs.

We thank L.C. Butler, T.J. Lee and W.T. Gilliam for their assistance in the gas chromatographic analysis of the hydrocarbon samples; C.R. Cripe and C. Somerville for their assistance in the design and implementation of specific experiments; C.R. Cripe, W.T. Gilliam, T. Dunn, K. Englert and B. Pierce for their help in implementation of the hydrocarbon field study; R. Montgomery, J. Clark, and Larry Goodman for their assistance in the toxicity studies. T. Maziarz for his assistance in processing and interpreting the experimental data from the field study; Carol Otey and Val Caston for typing and editing.

This report discusses the microbial degradation of hydrocarbon mixtures in aquatic systems, and the probable results of jet fuel leakage or spillage under various environmental circumstances. References to specific equipment or brand names should not be construed as endorsements of, or advertisements for those products.

This report has been reviewed by the Public Affairs Office (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This report has been reviewed and is approved for publication.

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TABLE OF CONTENTS

TABLE OF CONTENTS
(Concluded)

LIST OF FIGURES

LIST OF FIGURES
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LIST OF FIGURES
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LIST OF FIGURES
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# SECTION I

# INTRODUCTION

## A. OBJECTIVE

The objectives for this project were: (1) to simulate a spill of JP-4 fuel in a small estuarine pond and, by following the fate of selected hydrocarbons in the fuel, attempt to assess the value of laboratory experiments performed at the same time) in predicting events in the field study; (2) To determine if biotic or abiotic degradation of the synthetic missile fuels would occur in water and/or sediment and, if the fuels persisted, determine their toxicity to a standard test organism typical of marine habitats.

## B. BACKGROUND

Hydrocarbons are introduced into the surface waters of marine environments through runoff and erosion, effluent discharges, atmospheric deposition, and accidental spills. The extensive transportation of jet fuels over land and water, particularly in areas near bays, estuaries, and wetlands, has produced increased concern over the effects of these fuels on marine and estuarine environments (Reference 1). Of particular environmental concern in aquatic environments are the toxic hydrocarbons with the boiling range between the n-$C_6$ and n-$C_{14}$ hydrocarbons, a group that constitutes the major fraction of jet fuels. The degree of incorporation of these hydrocarbons into the water column of aquatic ecosystems, following a spill or discharge, will largely determine the potential for adverse environmental impact.

The fate of spilled hydrocarbons in aquatic environments is controlled by a variety of processes including surface spreading, evaporation, emulsification, and microbial and photochemical degradation. Sorption onto particulates can carry hydrocarbons, at varying rates, to bottom sediments. The importance of some of these processes on the fate of jet fuels has been addressed in a variety of laboratory studies (Reference 2). Because of our reliance on this laboratory information to make predictions about the fate of jet fuels in complex natural systems, comparisons of fate data from laboratory and field studies studies was necessary as a means of improving our confidence in the environmental significance of laboratory data.

As with jet fuels, the increased use of synthetic missile fuels creates a similar potential for spillage into estuarine and marine environments. A study of of the fate and toxicity of these fuels in aquatic test systems was therefore necessary.

## C. SCOPE

As with any laboratory studies that are potentially used to make predictions about events in the field, it is important to perform field validation of the results. In examining the laboratory information produced on the fate of jet fuels in aquatic systems, it was decided that the most important aspect of these studies to validate was the effects of sediment on biodegradation and volatilization rates. This decision was based on results which showed that when jet fuel was appled to the surface of the shallow water column contained

in the quiescent bottle test, it would evaporate at approximately the same rate as jet fuel spilled on the surface of a body of water in the field. There was little need of further validation. However, the fate of jet fuel when it becomes mixed with sediments is considerably more complicated because of the generally undisturbed nature of the sediment bed, the large volumes of water covering the sediment, and the metabolic potential (or lack of it) possessed by the microbial communities associated with the sediment. The ability to simulate these sediment factors in laboratory test systems was uncertain and thus field validation of this aspect seemed appropriate.

The quiescent bottle procedure incorporates an initial shaking step to promote contact of the jet fuel with sediment as it might occur during a spill incident. To artificially create a similar situation in the field, however, would require a major logistical undertaking that was beyond our field capabilities. It was decided, therefore, to perform a field validation exercise by initially contaminating sediments, from a selected field site, with a specified amount of jet fuel and then adding this contaminated sediment back to the field site. This procedure would essentially be equivalent to the initial shaking step and the subsequent incubation method in the quiescent bottle test. In addition, the contaminated sediment would be added to a laboratory test system. The fate of the hydrocarbons in both laboratory and field systems would be compared. Our studies have shown that information from laboratory tests was only partially sufficient for predicting the fate of JP-4 in a small pond. However, both laboratory and field studies indicated that many hydrocarbons in jet fuel will persist if they become associated with sediments in aquatic environments.

Since little information was known about the fate of missile fuel in aquatic systems, simple laboratory tests would be used to follow the disappearance rate of hydrocarbons in the missile fuels RJ-5 and JP-9. Shake-flask studies would be performed using water and sediment samples from three different estuarine sites. The importance of biodegradation relative to volatilization would be assessed by comparing sterile and nonsterile treatments. Toxicity of the missile fuels to microbial communities and mysid shrimp would also be examined using the endpoints of mineralization of radiolabeled substrates and mortality over a 96-hour period, respectively. Our studies have shown that the synthetic hydrocarbons of RJ-5 and JP-9 were quite persistent in water and sediment samples from aquatic systems, showing no biodegradation over the course of our experiments. The missile fuel, RJ-5 was toxic to mysid shrimp.

## SECTION II

## JET FUEL STUDIES

### A. RESEARCH PLAN

When fuel oils are spilled in aquatic systems, the associated hydrocarbons will either volatilize, dissolve in the aqueous medium, sorb to sediments and suspended particulates, or be degraded by communities of microorganisms. Our previous studies (Reference 2) have shown, based on laboratory tests, that these processes occur to varying degrees with jet fuel. To establish the validity and significance of these results, we elected to further study the fate of jet fuel using a field approach. The following plan of action was consequently developed:

a. Compare previous test results with published information.
b. Examine the literature for guidance in performing a field validation of laboratory data.
c. Design and implement a field study to assess the environmental fate of jet fuels in aquatic system.
d. Compare results of laboratory and field tests.

#### 1. Literature Review

Two important aspects of our previous laboratory studies merit special consideration because of their impact in assessing potential adverse effects of a jet fuel spill in an aquatic environment. These are: (a) the rapid biodegradation, in pristine waters, of aromatic hydrocarbons relative to aliphatic hydrocarbons when these hydrocarbons are available (i.e., not lost to volatility) to microbial communities in the water column and (b) the apparent persistence of jet fuel hydrocarbons when they become associated with natural sediments. The validity of these observations can be determined initially by making comparisons with the existing literature.

Relative biodegradation rates of aromatic and aliphatic hydrocarbons in petroleum products by natural microbial communities in aquatic environments is not clearly established and conflicting results appear in the literature. The general dogma is that normal alkanes are the first hydrocarbons to biodegrade in an oil spill situation; past reports have indicated that the aromatic fraction of oil was quite resistant to microbial attack (References 3 and 4). Lee et al. (Reference 5), for example, found that in Georgia salt marsh areas dosed with a heavy fuel oil, the aromatic hydrocarbons were not substantially degraded until approximately 45 days after the dosing. However, recent studies indicate exactly the opposite result. Fedorak and Westlake (Reference 6) examined the biodegradation of Prudhoe Bay crude oil under "shake-flask" conditions using a specific extraction and column-separation procedure to evaluate the relative biodegradability of saturated and aromatic hydrocarbons. They observed in pristine and polluted water samples that the aromatic fraction was degraded more rapidly than the saturate fraction. Supplementation with nitrogen and phosphorous increased the degradation of the saturate fraction in their experiments, but the simple aromatics (naphthalene, 2-methylnapthalene) still degraded more readily than the n-alkanes. Jones (Reference 7) revealed in the monitoring of oil-polluted sediments in the Shetland Islands that the

alkylaromatic hydrocarbons were also degraded before degradation of the normal alkanes. In fact, ratios of pristane to heptadecane remained relatively constant, while ratios of pristane to the aromatic components increased rapidly. Ward et al. (Reference 8), reported similar observations in a study of oil-polluted sediments from the Amoco Cadiz spill area. They observed a substantial decrease in the normal alkanes and the mono- and diaromatic hydrocarbons, but not the branched alkanes, triaromatics and dibenzothiophenes. Studies by Albaiges and Cuberes (Reference 9) showed that the degradation of alkanes was slower with increasing chain length, leading to relative persistence of the higher paraffins. The aromatic fraction of the oil they studied showed sequential, but relatively rapid, biodegradation; degradation of the alkylbenzenes preceded degradation of the polynuclear aromatic materials.

These results support our observations (Reference 2) of rapid biodegradation of many aromatic hydrocarbons but relatively slow degradation of the alkanes and branched alkanes, particularly those of higher molecular weight. This pattern of degradation may be due to the greater availability of the aromatics to the microbial communities in water, as many of the volatile aromatic hydrocarbons in jet fuel readily dissolve in the surrounding aqueous medium. This solution or water-soluble fraction (WSF) is dominated by alkyl benzenes and napthalenes (References 10 and 11). Researchers cannot agree on the factors affecting dissolution (increased availability to microorganisms) or evaporation (decreased availability to microorganisms) in any particular environmental sample. Boehm et al. (Reference 12) observed almost equal losses of aromatics (high solubility) and alkanes (low solubility), attributing their results to volatilization. Zurcher and Thuer (Reference 13), on the other hand, observed rapid loss of isopropylbenzene, compared to that for nonane, and attributed the results to dissolution. Several environmental factors have been shown to affect the WSF (Reference 11); lower temperatures decreased solubility of the WSF whereas water turbulence increased the WSF concentration. However, original composition of the oil or fuel was the dominant factor affecting the WSF. These variables, therefore, could greatly affect the observed rapid degradation of aromatic hydrocarbons.

The apparent persistence of hydrocarbons in fuel-contaminated sediments as seen in our previous work has been observed in other studies on oil degradation. Since petroleum hydrocarbons are hydrophobic, a major transport mechanism of these chemicals in riverine and estuarine systems is associated with suspended particulates. Pollutant hydrocarbons, therefore, tend to readily accumulate on particulate materials or in sediments (References 14 and 15). The fate of oil in natural sediments has been studied, using a technique of placing oil-contaminated sediments in plexiglass trays and lowering the trays onto the sediment bed of a particular field location (References 16 and 17). Extensive sampling over a 2-year period revealed very slow biodegradation. A significant decrease in the aliphatics (less than $C_{17}$ length) and diaromatics was observed, but little decrease in the triaromatics and sulfur-containing hydrocarbons occurred. Although attempts were made to mix oil and sediments in these studies, a high degree of spatial and temporal variability was noted between replicate samples because of sediment trapping of the oil. Hydrocarbons from refinery wastes entering an estuary in England were found to rapidly adsorb to sediments (References 18 and 19). Little change in concentration of hydrocarbons with depth in the sediment suggested that mixing or biodegradation was not occurring. Hanes and Atlas (Reference 20) examined the biotic fate

of Prudhoe Bay crude oil in near shore sediments of the Beaufort Sea, using a small enclosure in the field. Oil in sediments degraded very slowly, with only the low molecular weight (< C18) alkanes showing significant losses over a 1-year exposure period.

In other situations, significant biodegradation of hydrocarbons in sediments has, in fact, been detected or inferred. Jones et al. (Reference 7) and Ward et al. (Reference 8) found that alkanes and aromatic hydrocarbons degraded in sediments following contamination from an oil spill. Bates et al., and Hamilton et al. (Reference 21 and 22), having found three times the concentration of polynuclear aromatic hydrocarbons in suspended sediments as they did in surficial bottom sediment, concluded that because the flux of aliphatic hydrocarbons to sediment was greater than the rate of hydrocarbon accumulation in sediments, biodegradation of sorbed hydrocarbons was occurring. Others have provided indirect evidence for hydrocarbon biodegradation in sediments by showing that the number of hydrocarbon degraders was elevated in oil-contaminated sediments relative to uncontaminated sediments (References 23 and 24). Four different types of oil (Arabian, Libyan, Number 6 and Number 2 crude oil), when applied to salt marsh test plots, persisted in sediments for up to 1 year (Reference 25). However, enrichment of alkyl-substituted phenanthrene relative to alkyl-substituted naphthalene (present in the original oils) and the enrichment of isoprenoid hydrocarbons (pristane and phytane) relative to n-alkanes suggested that some degradation had occurred. Biodegradation of hydrocarbons in kerosene in sediment-water slurries taken from lakes with differing histories of hydrocarbon pollution has also been demonstrated; the highest degradative activity was associated with the most oil-polluted samples (Reference 26).

These results suggest that, in certain environmental situations, sediment-associated hydrocarbons are very slow to degrade. It supports our observations (Reference 2) of slow biodegradation of jet fuel hydrocarbons in sediments. Lack of degradation is probably due to a variety of environmental factors but the availability of dissolved oxygen, a factor shown many times to be mandatory for biodegradation of fuel hydrocarbons, is the most likely restriction for biodegradation in the sediments.

The ecological consequences of this persistence are illustrated in the outdoor tank studies carried out by a research group at the University of Rhode Island. These studies dealt with the effects of crude oil on a coastal marine ecosystem using large outdoor tanks (References 27, 28, and 29 30). The incorporation, distribution and fate of Number 2 fuel oil in sediments has been studied in these tanks. About 50 percent of the insoluble saturated hydrocarbons were adsorbed to suspended particulates, which eventually were deposited on the sediment bed and slowly mixed into the sediment by bioturbation. Biodegradation of the oil in sediments occurred, but a residue of 12-20 percent of the original Number 2 fuel oil persisted for at least 1 year. Chronic (5-month continuous), low-level (range: 60-350 parts per billion) dosing of the tanks with the Number 2 fuel oil resulted in significant toxic effects to both the planktonic and benthic communities (Reference 27). Phytoplankton populations increased in biomass and had a radically different species composition relative to control tanks. The observed changes were probably a result of decreased grazing pressure caused by toxicity of the fuel to the zooplankton and benthic suspension feeders. Likewise, benthic protozoan

populations in the sediment increased dramatically as a result of substantial decreases in predatory macrofaunal populations. Bioturbation activities in the sediments were also reduced due to the drop in macrofaunal numbers.

Most important from the standpoint of microbial activity, was a major suppression in the production of total inorganic nitrogen ($NH_4$, $NO_2$, $NO_3$) from the sediments. Considering that the principal growth-limiting factor for phytoplankton productivity in Narragansett Bay is controlled by the rate of mineralization of organic nitrogen deposited from the water column into the sediment and by the eventual efflux of nitrogen from sediments, this effect is quite serious and represents an adverse effect that could be experienced in any coastal ecosystem.

Thus, our observed persistence of jet fuel hydrocarbons in laboratory test systems with sediment (Reference 2) is cause for concern because of the potential ecological impacts. However, before this concern can be factored into an environmental management concept that might be used by the Air Force in the event of a jet fuel spill, further validation of the observations must be undertaken. A field-dosing experiment with jet fuel was therefore selected as a means of further validation.

2. Field Dosing Methods and Rationale for Field Study

Field studies relating to hydrocarbon degradation in aquatic systems are generally of three types: examination of environments presently polluted with hydrocarbons (spills, effluents, etc.), dosing of large environmental enclosures, and dosing of laboratory microcosms. Monitoring of the Searsport, Maine oil spill (1971), for example, has shown that lower temperatures and anoxic conditions greatly slowed the weathering of the oil hydrocarbons (Reference 31). Similar conclusions were reached in an in situ study of the fate of Metula oil spilled on the beaches bordering the Straits of Magellan (Reference 32). Biodegradation of oil from the Amoco Cadiz spill was found to be very slow, possibly because of inorganic nutrient limitations (Reference 33). The monitoring of oil-polluted sediments in the Shetland Islands (Reference 7) showed that degradation occurred primarily in aerobic sediments and that the alkylaromatic hydrocarbons were degraded before normal alkanes. Others have examined the fate of petroleum hydrocarbons in salt marsh areas by dosing a field site directly (References 5, 17, and 33). Fusey and Outdot (Reference 34), after having dosed sediments in the field with light crude oil, were able to develop a tentative semiquantitative graphic model to evaluate the relative parts physical removal and biodegradation play in the decontamination of oil-polluted seashore sediments.

The dosing of large environmental enclosures has frequently been used for fate studies of hydrocarbons. The vertical mass fluxes and sedimentation rates of crude oil have been studied in large flexible plastic containers of water from the North Sea (References 35, 36, 37 and 38). Adsorption to and subsequent sedimentation of plankton and organic detritus caused rapid sinking of the petroleum hydrocarbons and comparatively little microbial mineralization was observed. Photo-oxidation of the hydrocarbon components, possibly to toxic polar products, was also observed in the enclosure experiments (References 35 and 38). We have previously mentioned the

use of large outdoor tanks to study the fate and effects of oil (References 27). The tanks, which comprise a physical simulation model of Narragansett Bay, are 5.5 meters high, 1.83 meters in diameter, and contain a box core of sediment (30 cm deep) that continuously receives unfiltered bay water (turnover time equal to 27 days) while exposed to natural light and temperatures regimes. Stirring devices were designed to direct turbulent energy onto the sediment to effect resuspension of flocculant material as observed in the bay. The size of these tank systems, although originally designed to satisfy a specific sampling regime, greatly restrict experimental manipulation. These systems represent some of the most extensively characterized long-term enclosure-type experiments to date, providing detailed descriptions of annual plankton and nutrient cycles, benthic invertebrates, turbulent mixing, and replicability.

Recent oil degradation studies in microcosms have produced some interesting hypotheses about the fate of oil in aquatic systems. Circular glass containers, filled with water overlying a sediment bed, were employed by Albaiges and Cuberes (Reference 9) to show that photolysis and biodegradation play a very integrated part in the fate of oil, particularly in terms of the degradation of polyaromatic hydrocarbons. They also questioned the widely accepted dogma that n-alkanes are metabolized more rapidly than either naphthenes or aromatics; these hydrocarbon families, in fact, showed a very nonuniform reactivity throughout their complete molecular range. Horowitz and Atlas (Reference 39) also observed, in a unique continuous flow-through microcosm, that most components in their test oil degraded at about similar rates, again questioning the relative reactivity of the metabolic transformations of petroleum hydrocarbons in complex natural systems. A microcosm designed to model the fate of oil in a tidal flat system typical of the Wadden Sea (Reference 40) revealed that bioturbation by benthic invertebrates caused the oil to be rapidly buried in the sand. The apparent lack of degradation in the sediment resulted in the persistence of oil and subtle long-term toxicological effects to the biota contained in the microcosm. Franco et al. (Reference 41) also showed similar ecosystem-level effects in a pond microcosm exposed to coal-derived oil. The toxic response they observed was not an effect that could be easily observed in more simplified types of laboratory bioassay tests.

Based on information in these reports, it was decided that direct dosing of a field pond would provide the most practical and efficient means of field-validating our laboratory results. The rationale was that field enclosures were too mechanically complex and expensive and that microcosm studies were complicated because of a need to simulate appropriate water depth and natural dissolved oxygen concentrations. In addition, it was decided that application of jet fuel to the water surface of a pond without accompanying water turbulence or mixing would result in rapid volatilization of the fuel with little residual for analysis of hydrocarbon fate. Mechanical mixing of a pond to enhance exposure of the fuel to sediments, as might be expected under certain spill situations, was considered but was felt to be unsafe because of the need to use powered mixing devices in an area of flammable hydrocarbon vapors. We also knew, that vigorous mixing would excessively disrupt the natural state in a pond, particularly any low oxygen zones. However, much of our information from laboratory tests dealt with close association of fuel hydrocarbons with sediments: i.e., quiescent bottle tests were initially shaken

for 1 hour to vigorously mix the fuel into sediment-water slurries (Reference 2). Therefore, a field-dosing method had to be developed which would assure significant exposure of the jet fuel to sediments.

3. Experimental Plan for Field Dosing with JP-4

The field pond was dosed by direct contamination of sediments; the protocol (Figure 1) was to physically remove sediments from a pond, thoroughly mix them with a specified amount of jet fuel, and add the contaminated sediments back to the pond. By periodically removing sediment samples and extracting with solvent, we could quantitate changes in hydrocarbon composition by gas chromatography. We reasoned that when the jet fuel-sediment mixture was added to the pond, much of the fuel would initially evaporate, but enough residual hydrocarbon would remain such that its longer-term fate could be assessed. If hydrocarbons persisted in sediments, as had been observed in laboratory tests, this should be apparent in the pond.

The addition of oil-contaminated sediments to aquatic systems as a means of studying the fate of hydrocarbons in natural sediments was successfully employed by Haines and Atlas (Reference 17) in their work on the in situ microbial degradation of Prudhoe Bay crude oil in sediments. They collected sediment, mixed it with crude oil (5 percent volume for volume) until no visible oil slick accumulated on the sediment surface and then dispensed the mixture into 25 by 25 by 5 cm plexiglass trays. The trays were then replaced on the bottom of a lagoon and incubated for up to 2 years. This method allows water to freely exchange with the sediment in the tray and thereby replenish oxygen and inorganic nutrients. But it prevents the contaminated sediment from being mixed with uncontaminated sediment and thereby slows dilution of residual hydrocarbons. At each sampling period in their study, a tray was brought to the surface, with minimal disturbance, and the residual oil content of the sediment was determined. In their case, very little biodegradation of the oil occurred because of cold temperatues in the test area.

The application of this method to our field study seemed very appropriate. As a result, a procedure was developed (see Figure 1) in which sediments contaminated with JP-4 were placed in plexiglass trays and set out on the bed sediment of a pond. However, rather than removing the trays for sampling, we sampled the trays in place using a simple suction sampling device. In addition, we placed the JP-4/sediment mix directly on the sediment bed (no trays) to determine the fate of the jet fuel under conditions where dilution into uncontaminated sediment was not restricted. Finally, the JP-4/sediment mix was used in a standard quiescent-bottle test, as previously described by Spain et al. (Reference 2), in Figure 1, to determine the relative rates of hydrocarbon in jet fuel under sterile (no biodegradation) and nonsterile (biodegradation) conditions.

The fate of the volatile hydrocarbons in sediments depends to a large extent on the height of the water column over the sediments (i.e., controls volatilization) and the amount of oxygen available (i.e., controls biodegradation). In a pond, both conditions will be variable; areas near the center of a pond will be the deepest (less volatilization) and may also have zones with lowest dissolved oxygen (less biodegradation). Thus, the fate of JP-4

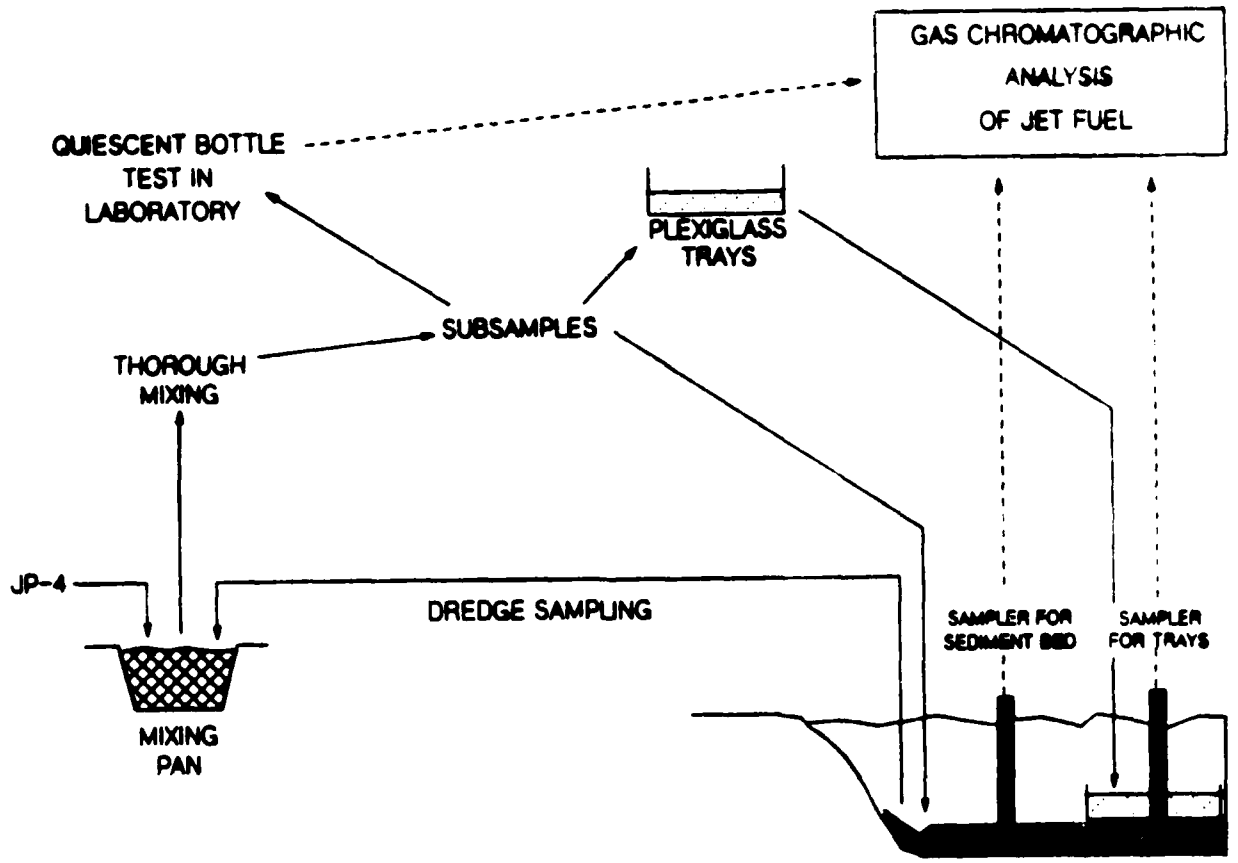



Figure 1. Schematic Diagram of Experimental Design for Jet Fuel Study

may differ significantly, depending on its location in a pond. To accomodate these possibilities, our field dosing was carried out by placing JP-4 contaminated sediments (in trays and directly on the sediment bed) in a deep and shallow area of the pond. Accordingly, a second set of quiescent bottle tests was set up with larger amounts of water covering the sediments to roughly model the deeper portion of the pond.

4. Characteristics of Field Site

The pond selected for our field test was located on Santa Rosa Island at an area designated as Big Sabine Point (Figure 2) and had a surface area of approximately 160 $m^2$ (Figure 3). A small sand dune limited exchange of the pond with Santa Rosa Sound to spring tides and storms. Site B, our primary shallow water site (approximately 1-1.5 meters deep) had a relatively stable concentration of dissolved oxygen, averaging around 6 ppm (Figure 4). Salinity, pH, and temperature were relatively constant. Site C, our primary deep water site, had dissolved oxygen concentrations which frequently dropped below 1 mg/$\ell$ at the lower depths (Figures 5,6,7). Changes in pH and salinity for this site were relatively small and the values were similar to those at Site B. Temperature fluctuated over an 11° C range with time but showed only a slight decrease in temperature with depth (no stratification). Measured values of these four parameters at sites B and C are given in Appendix A.

The sediment at Site B consisted of sand mixed with organic detritus (plant and algal remains) while the sediment at Site C consisted of sand layered with a mat (1-4 cm) of light fluffy organic detritus. Differences in water levels in the pond, which varied with the tides and the weather conditions, were generally less than 25 cm.

Dye studies, using Rhodomine WT, were carried out to measure water turnover rates. Dye was added to the water and mixed for 15 minutes with a trolling motor. Water samples (5 m$\ell$) were taken at Sites B and C at different depths and brought back to the laboratory to measure the fluorescence using a Turner Fluorometer. Relative changes in dye concentrations are given in Appendix A. In general, dilution was slow enough that it would not dramatically affect hydrocarbon disappearance rates. Very little difference in concentrations of dye with depth was observed indicating uniform mixing within the water column.

B. METHODS

1. Dosing

Sediment, which was largely organic detritus mixed with small amounts of sand, was collected from the pond bottom using an Eckman dredge. A fiberglass box (90 cm by 90 cm by 40 cm) was filled with the sediment (as a thick slurry) and JP-4 fuel added. The amount of fuel added was based (small scale laboratory tests) on adding the maximum amount of fuel without leaving significant amounts of floating oil droplets, i.e., all adsorbed to sediments. The jet fuel-sediment mixture, after thorough mixing, was first added, using a bucket, back to the pond at Sites B and C. We attempted to distribute the contaminated sediments as evenly as possible over a 1.2 - 1.5 square meters area of pond bottom at each site. Second, the contaminated sediments were

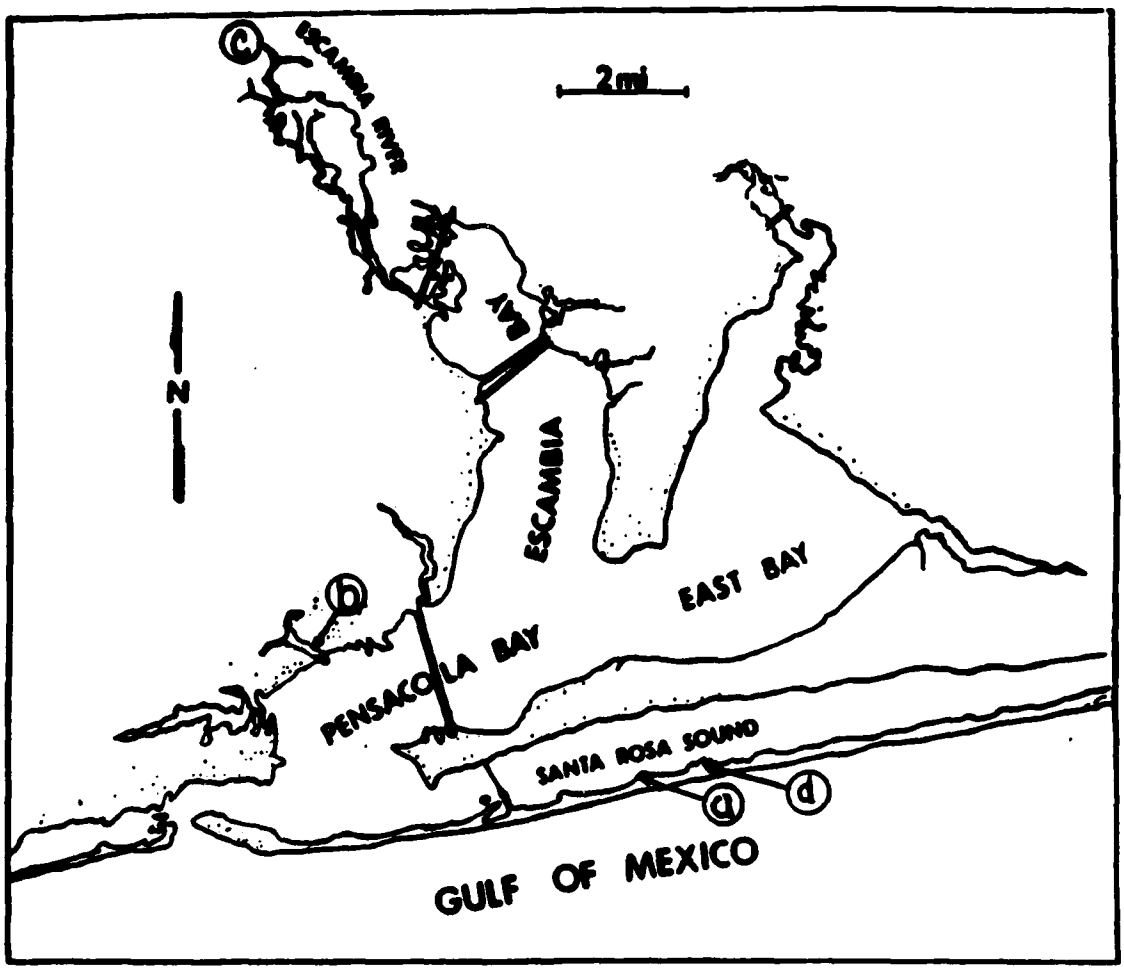



Figure 2. Escambia Bay Estuary Showing Sampling Sites: a. Range Point, b. Bayou Chico, c. Escambia River, d. Big Sabine Point.

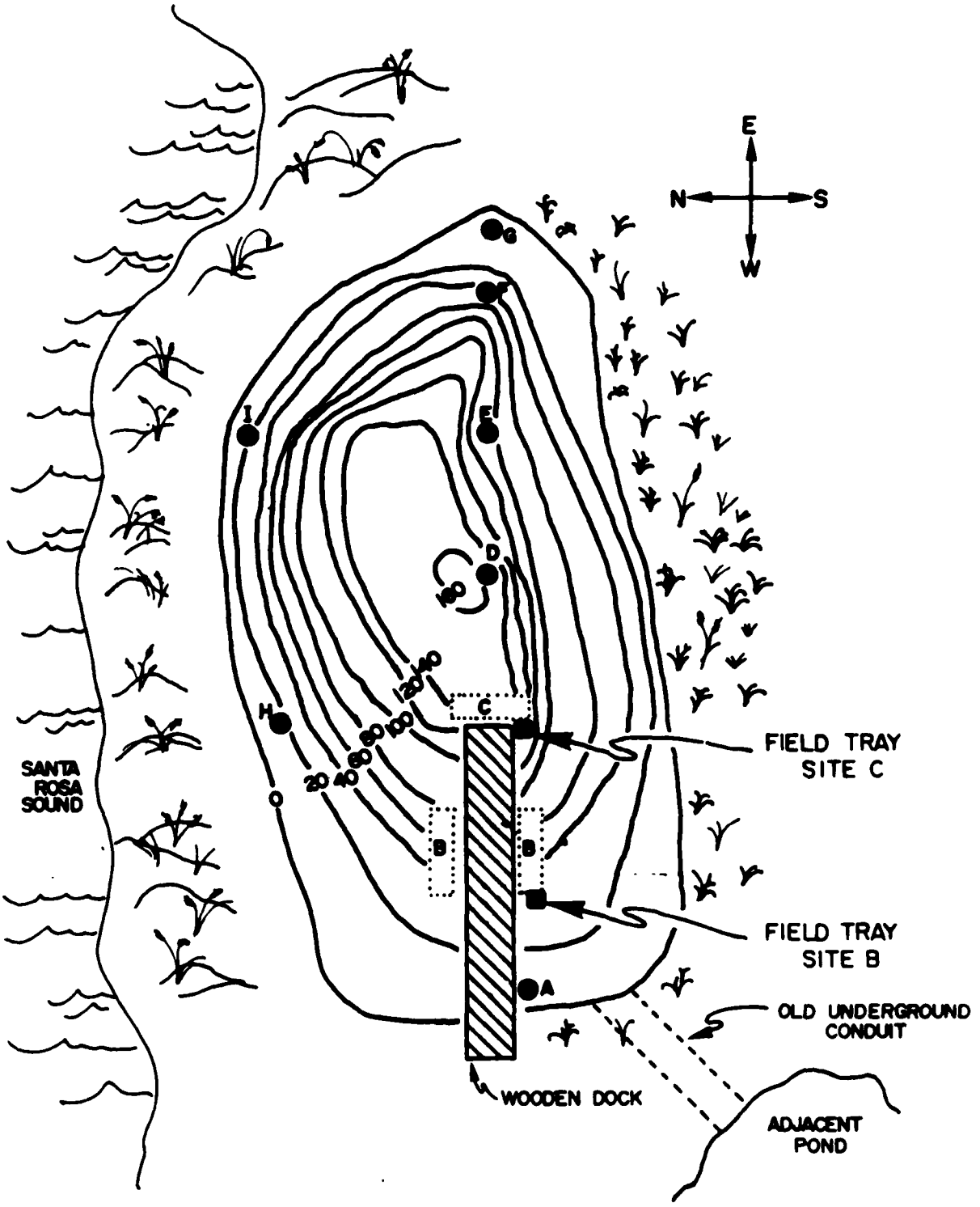



Figure 3. Topographic Map of Study Pond. Depths Are in Inches. The Pond was Surrounded by Rooted Vegetation.

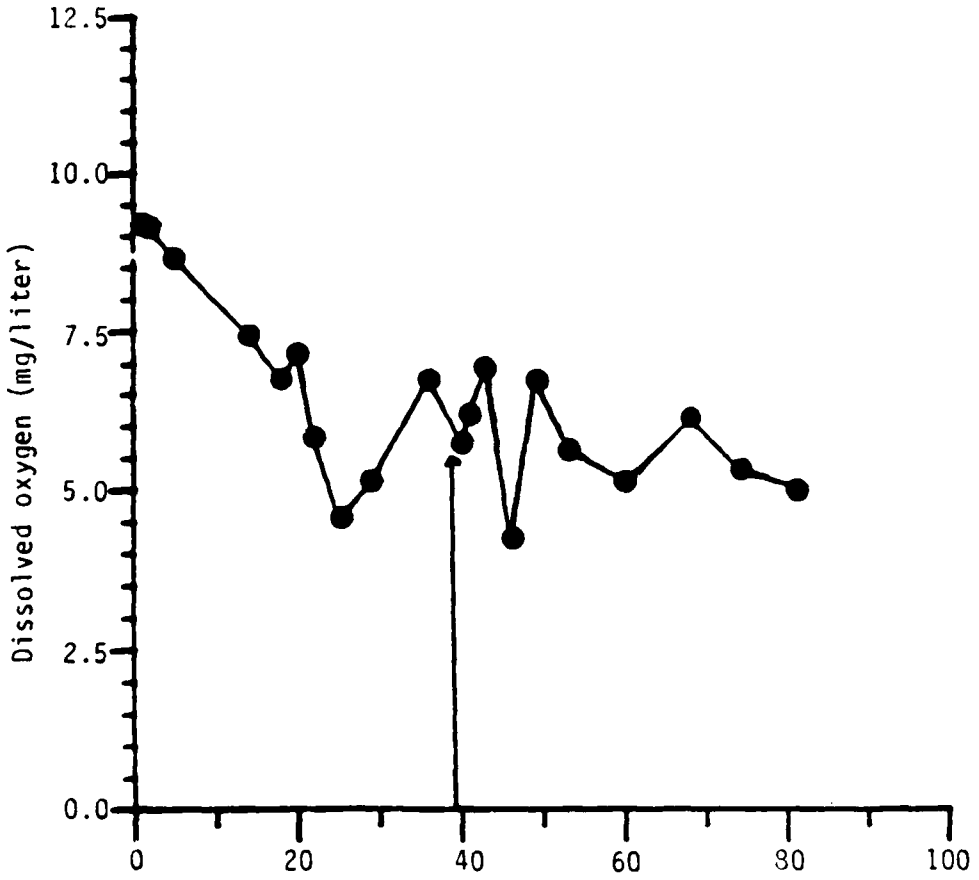



Figure 4. Concentrations of Dissolved Oxygen Measured Approximately 15 cm Below the Water Surface at Site B (Shallow Water Site) in Test Pond. Arrow Indicates Time of Dosing with JP-4.

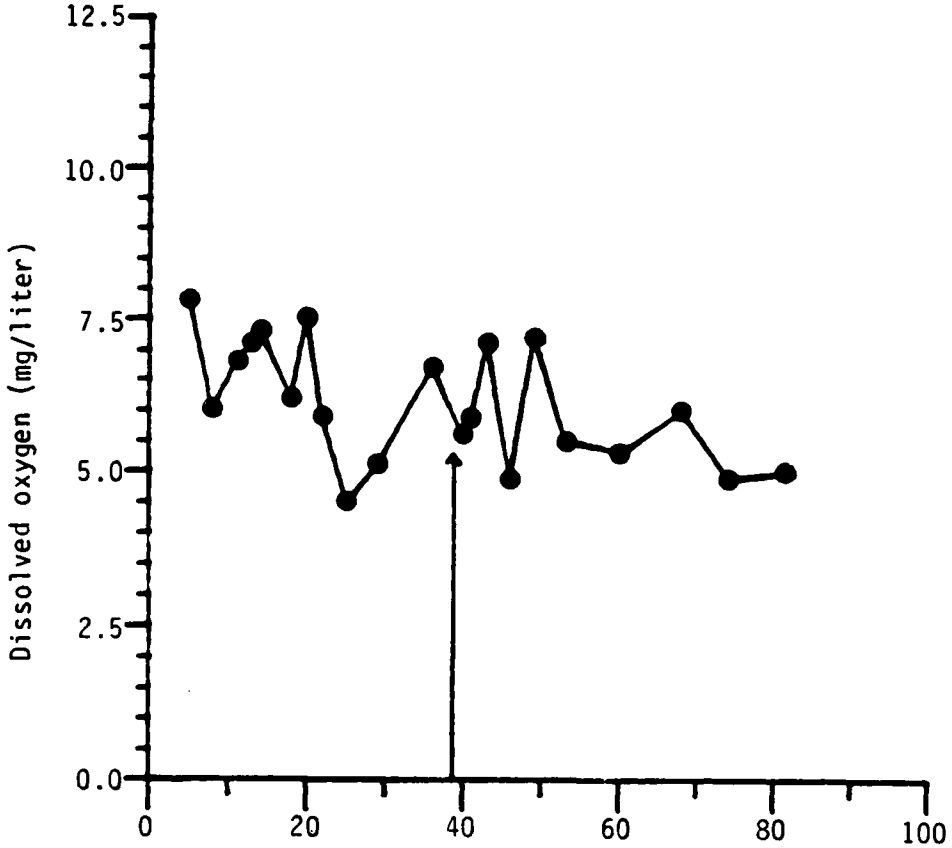



Figure 5. Concentrations of Dissolved Oxygen at Site C (Deepwater Site) in the Test Pond at Approximately 15 cm Below the Water Surface. Arrows Indicate Time of Dosing with JP-4 Contaminated Sediment.

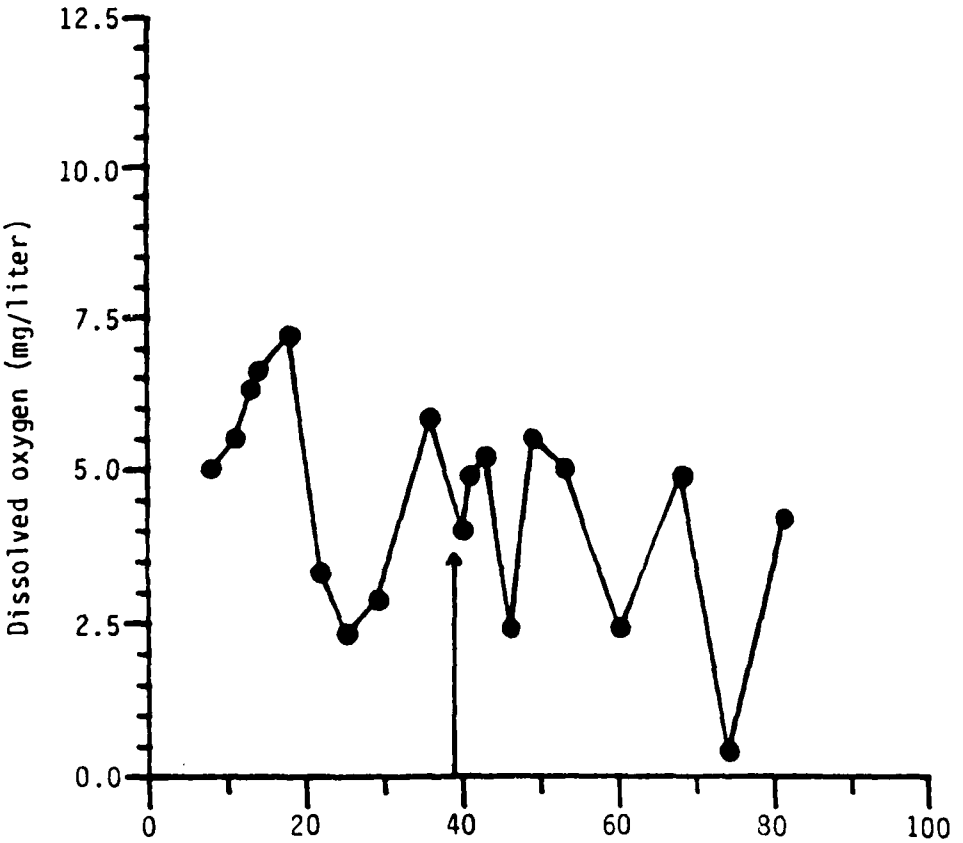



Figure 6. Concentrations of Dissolved Oxygen at Site C (Deepwater Site) in the Test Pond at Approximately 175 cm Below the Water Surface. Arrows Indicate Time of Dosing with JP-4 Contaminated Sediment.

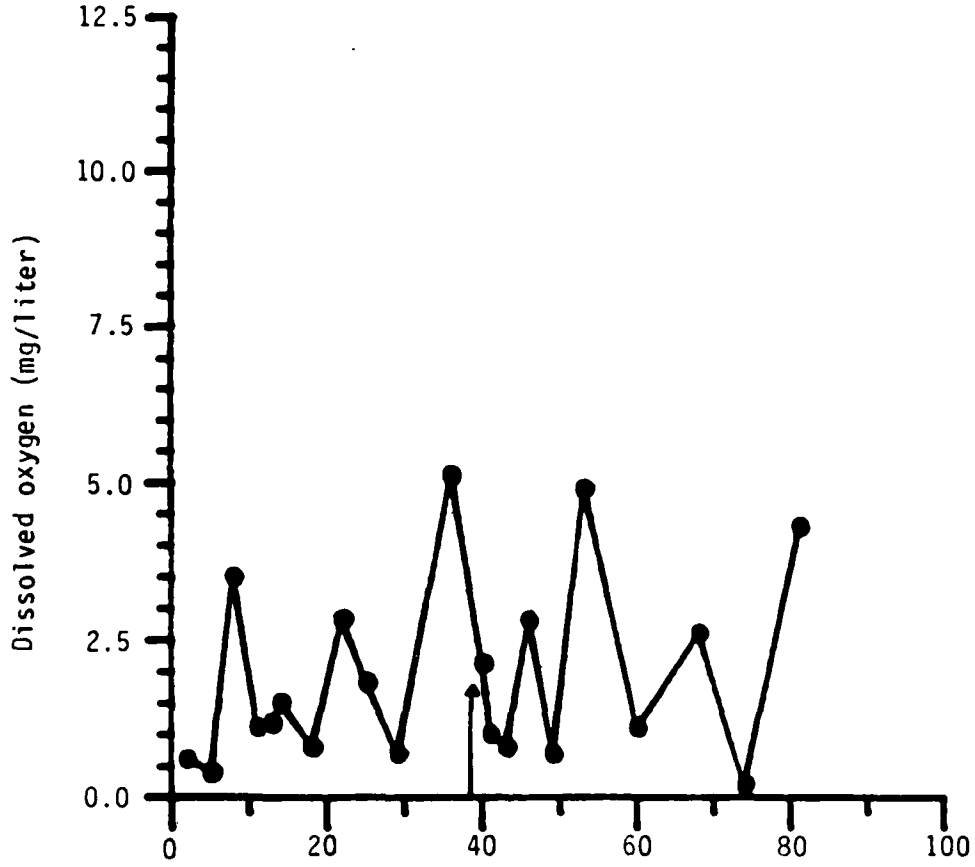



Figure 7. Concentrations of Dissolved Oxygen at Site C (Deepwater Site) in the Test Pond at Approximately 350 cm (Sediment-Water Interface) Below the Water Surface. Arrows Indicate Time of Dosing with JP-4 Contaminated Sediment.

added to plexiglass trays 30 cm by 30 cm by 4 cm high to give a sediment depth of approximately 2 cm. The trays were then lowered, without disturbing the sediment inside, to the pond bottom at Sites B and C.

2. Sampling

Water samples from the pond were taken with a clean bottle submerged approximately 25 cm below the water surface. Sediment samples were taken as follows: five glass coring tubes (3.5 cm in diameter) were inserted into the bottom sediment at the chosen sampling site. A small glass tube, stoppered at one end, was inserted (open-end first) into the water column contained within the coring tube. When contact was made with the sediment, the stopper was released; water rushing into the tube sucked up the light fluffy detrital material contained within the core, leaving the sand behind. The smaller tube was restoppered, and withdrawn; the sediment slurry inside was released into a clean sample bottle. Several resamplings in the core tube removed most of the detrital material. These were combined in the same sample bottle. The slurry in the sample bottle was allowed to settle and the water decanted to produce a thick sediment suspension. All five of the core tubes were sampled in a similar manner and analyzed separately. Sampling at Sites B and C was set up in a grid pattern to avoid sampling the same spot twice.

The trays were sampled by placing the open end of a glass tube (1.5 cm diameter) stoppered at one end, into the water until contact with the trays was made. Water rushing into the tube after the stopper was removed, sucked in some of the fuel-contaminated detrital material in the tray. The tube was restoppered and removed from the water; the sediment slurry contained inside was released into a sample bottle.

3. Bottle tests

Modifications of the standard quiescent bottle test (Reference 2) were used to provide laboratory information on the biotic and abiotic fate of JP-4 in water and sediment systems. Rather than adding the jet fuel to the water or water-sediment slurries in the bottles and shaking for the initial 1-hour period as described previously, the fuel-contaminated sediment used in the field dosing was added (5000 mg/l) directly to the bottles. In one set of bottles the sediment was covered with 25 mLs of pond water and the bottles incubated on their sides without closures as described previously. These were referred to as "shallow water" tests and were, therefore, simulated models for Site B in the pond. Sterile systems received 2 per cent formalin.

Another set of bottles was prepared containing the fuel-contaminated sediment covered with 150 mLs of water which had been deoxygenated by sparging with nitrogen. The bottles were incubated upright to reduce surface area available for volatilization and reoxygenation. These bottles modeled Site C in the pond, i.e., relatively large water depth over sediment and reduced oxygen conditions.

Bottle tests were run in duplicate and hydrocarbon concentrations were reported as means. Variation in concentrations among duplicates was generally less than ± 10 percent.

4. Chemical analysis

Water and sediment slurry samples were extracted with methylene chloride (sample: solvent ratio of 10:1) in 50 mℓ Teflon®-lined screw-capped test tubes for 1 hour by shaking in a rotary shaker. The resulting emulsion was broken by centrifuging the samples for 30 minutes at approximately 3000 rpm. Subsamples of the methylene chloride were then injected by auto sampler (H.P. 7671) into a capillary gas chromatograph (H.P. 5735) using the following conditions;

| | |
|---|---|
| Column: | 60 meters by 0.25 millimeter inside diameter with 1.0 micrometer bonded SPB-1 (Supelco, Inc.) |
| Carrier: | Hydrogen |
| Detector: | Flame ionization detector at 350 degrees centigrade |
| Injection: | 1.5 microliters splitless |
| Injector Temp: | 250 degrees centigrade |
| Oven Temp: | Programmed: 70 degrees centigrade for 2 minutes., ramp at 2 degrees/minute to 190 degrees centigrade 8 minutes hold |

Quantitations were carried out by computer evaluations of peak retention times and peak area integrations with a Hewlett-Packard 3357 LAS data system. Thirty-three hydrocarbons within the large number of components in the jet fuel were selected for quantitation. These compounds represented the major constituents by weight. Each compound shown was quantitated by comparison to a standard mixture formulated from analytical standards of each compound. To reduce the possibility of nonlinear detector sensitivity extrapolations, the concentration of each standard in the mix was proportional to that found in JP-4.

Quality assurance procedures consisted of checks on extraction efficiency, interfering peaks and spiked recoveries. Fifty tubes of uncontaminated sediment (5 mℓs of slurry sample per tube) from the pond were kept frozen until use at the appropriate sampling time. For each set of jet fuel-containing sediment samples taken from the field for analysis, two tubes of uncontaminated sediment were thawed. One tube was extracted as described above and the extracts were checked for gas chromatographic background peaks which might interfere with the hydrocarbon analysis. None were found in any of the samples. The second tube was spiked with 50 μℓ of jet fuel, shaken in a screw-cap test tube for 1 hour and then extracted and analyzed as above. Extraction efficiencies for total hydrocarbon concentration were generally greater than 90 percent; efficiencies for individual hydrocarbons were better (> 95 percent) for the higher molecular weight alkanes and these were used as a guage for extraction efficiency. Larger variability (± 15 percent) in extraction of the lower molecular weight hydrocarbons was probably due to the volatility of these components during sample handling.

Likewise, 50 jet fuel-contaminated sediment samples (5 mLs of slurry sample per tube) were frozen at the beginning of the experiments. During

each analysis of samples from the field, replicate tubes of the contaminated sediment were thawed and extracted as above. Recoveries were as good as the spiked sediment samples, indicating that long-term (2 months) contact of the jet fuel with sediment did not reduce extraction efficiency. Variation in total hydrocarbon concentrations from replicate sample extractions was less than ± 15 percent; ratios of hydrocarbons to tetradecane concentrations were always within ± 5 percent.

Duplicate samples were periodically taken from the sediment bed in the field and from the plexiglass trays. Considerable variations in concentration were observed as expected because of the patchiness in juet fuel distribution. The sample with the highest tetradecane and/or pentadecane concentrations was used. Where only single samples were taken, if the tetradecane concentration was below 10 percent of the calibration standard, the samples were discarded.

5. Quantitation of hydrocarbon disappearance

Disappearance of hydrocarbons in the jet fuel was determined by examining the ratio between the concentration of any hydrocarbon of interest with the concentration of another hydrocarbon in the jet fuel which was known to disappear very slowly, or not at all (i.e., a conservative tracer) over the time course of the experiment. This method is commonly used for assessing the fate of hydrocarbons in natural samples (References 7, 25, 32, 33); for example, ratios have been developed with the branched hydrocarbons, pristane and phytane, since they are some of the slowest to biodegrade, volatilize or disappear by dissolution in the environment (Reference 25, 7). Tetradecane was selected as the conservative tracer for jet fuel because in laboratory tests this hydrocarbon was consistently one of the slowest to disappear, either through volatility or biodegradation relative to the other hydrocarbons. By using the ratioing method, the amount of jet fuel sampled was not critical, as long as enough jet fuel was present for gas chromatographic analysis.

Based on the quality assurance procedures described in the methods section, we established that hydrocarbon ratios below 10 percent of the standard were generally not clearly different from zero, although when a particular hydrocarbon continued to show a slight gas chromatographic peak at each sampling time, small amounts of hydrocarbon appeared to remain in the sample. Differences in hydrocarbon ratios that were not greater than ± 10 percent were considered as unchanged.

C. RESULTS

1. Dosing

Upon addition of the contaminated sediment to the pond, a large amount of jet fuel rose to the water surface, shown by an obvious oily sheen. Much of the fuel on the water surface evaporated within 1-2 hours after dosing, while most of the fuel odor associated with the dosing also disappeared. Concentrations of hydrocarbons in the water column of the pond following dosing were below detection limits in 1 liter samples. A small amount of fuel remained around the animal cages used for the toxicity study. Small sheens of fuel continued to migrate from the cages for several hours.

Suspended sediment in the pond following dosing quickly settled out. Indigenous fish and crabs showed no overt signs of stress as a result of the dosing.

2. Trends in hydrocarbon disappearance

Actual concentrations of 33 selected hydrocarbons measured in sediment samples taken from the bottle tests, the plexiglass trays, and the field are given in Appendix B. Sampling times were adjusted to prevent backlog of samples. Tetradecane, the selected conservative tracer, was present in all samples and was one of the slowest to disappear. Thus, the concentration over time of all other hydrocarbons was measured relative to the concentration of tetradecane. Tables of ratio values with tetradecane for all 33 hydrocarbons analyzed are also given in Appendix B. Values are expressed as percent of standard: i.e., the ratio of any hydrocarbon to tetradecane in fuel-contaminated sediment before it was used for dosing (the standard) was set to 100 percent.

Figures 8 through 61 show the change in hydrocarbon ratios over time in the shallow water (Site B) and deep conditions (Site C), respectively for samples from the bottle tests (sterile and active), plexiglass trays, and pond sediment. Data points at t = 0 are based on analyses of the fuel-contaminated sediment before its addition to the test systems. A rapid initial disappearance of the hydrocarbons relative to tetradecane was observed in all samples. We believe this initial loss was due largely to volatilization and dissolution of the hydrocarbons during addition of the fuel-contaminated sediments to the bottles, plexiglass trays and the pond. Initial decrease in the ratios was generally much less in the pond sediment samples. To compare hydrocarbon losses in the various test systems, only decreases in the ratios beginning after Day 2 were generally considered. The following summarizes the general trends observed in the data.

a. Bottle Tests

Data from the bottle tests are the only information that can be used to assess the quantitative aspects of disappearance (sampling was precise and consistent) and the actual contribution of biodegradation (differences in sterile and active systems).

Hydrocarbon disappearance was generally smooth and steady: i.e., variability in sampling and analysis was quite low; repeated analysis of frozen samples of contaminated sediment gave consistent (less than $\pm$ 10 percent variation) recovery. In many cases, hydrocarbon ratios in samples from sterile bottles increased with time rather than the expected lack of change or decrease. If hydrocarbons become more completely sorbed with time into the sediment matrix (diffusion into the interstitial space of the organic matter on the sediment surfaces), as we have observed in other experiments, their tendency to be lost by volatilization during sample workup will be reduced; i.e., extraction efficiency will appear to increase. This may account for the increase in hydrocarbon ratios in sterile bottles over time. The higher molecular weight hydrocarbons, which are less volatile, would consequently be less affected by the sample workup and sorption, and ratios with these hydrocarbons showed less of a tendency to increase with time.

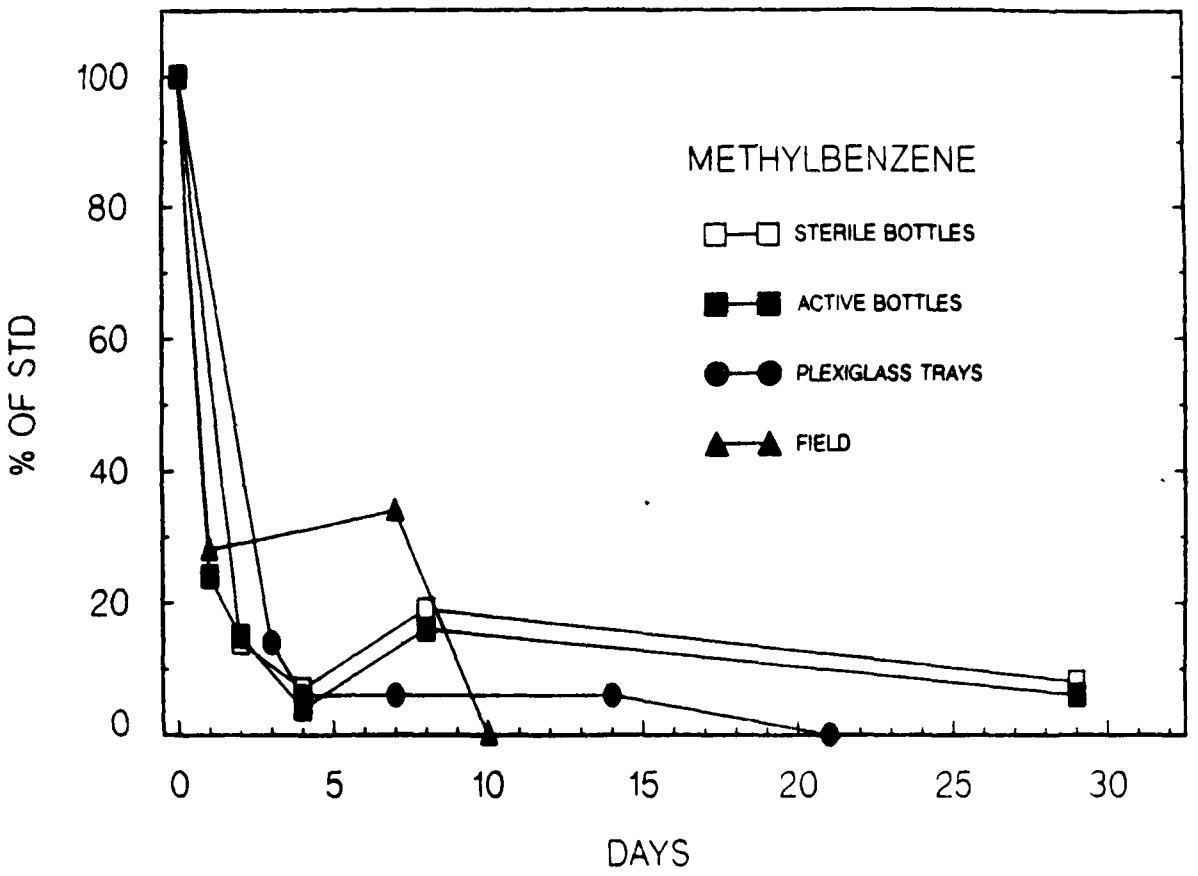



Figure 8. Change in Concentration Ratio (Expressed as Percent of Standard) of Methylbenzene to Tetradecane in Samples Taken from the Shallow Water Systems.

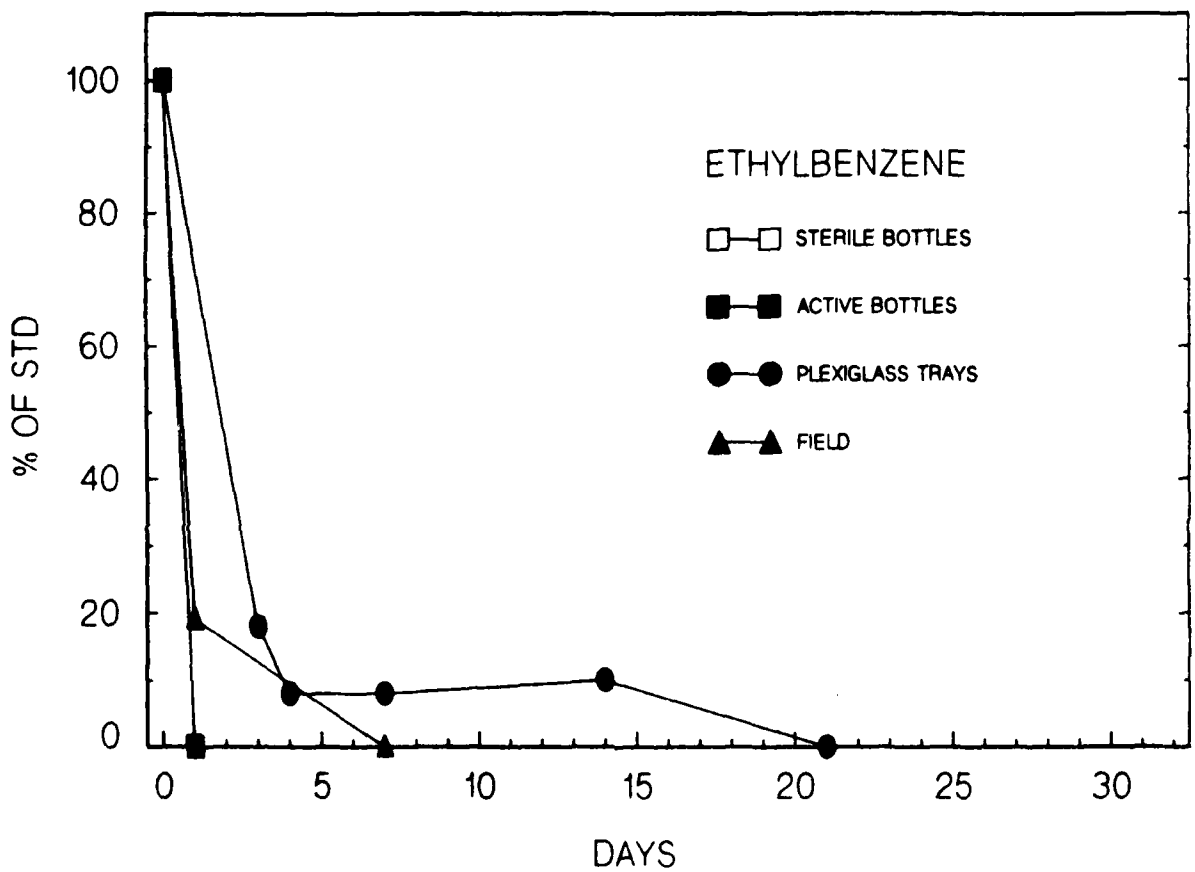



Figure 9. Change in Concentration Ratio (Expressed as Percent of Standard) of Ethylbenzene to Tetradecane in Samples Taken from the Shallow Water Systems.

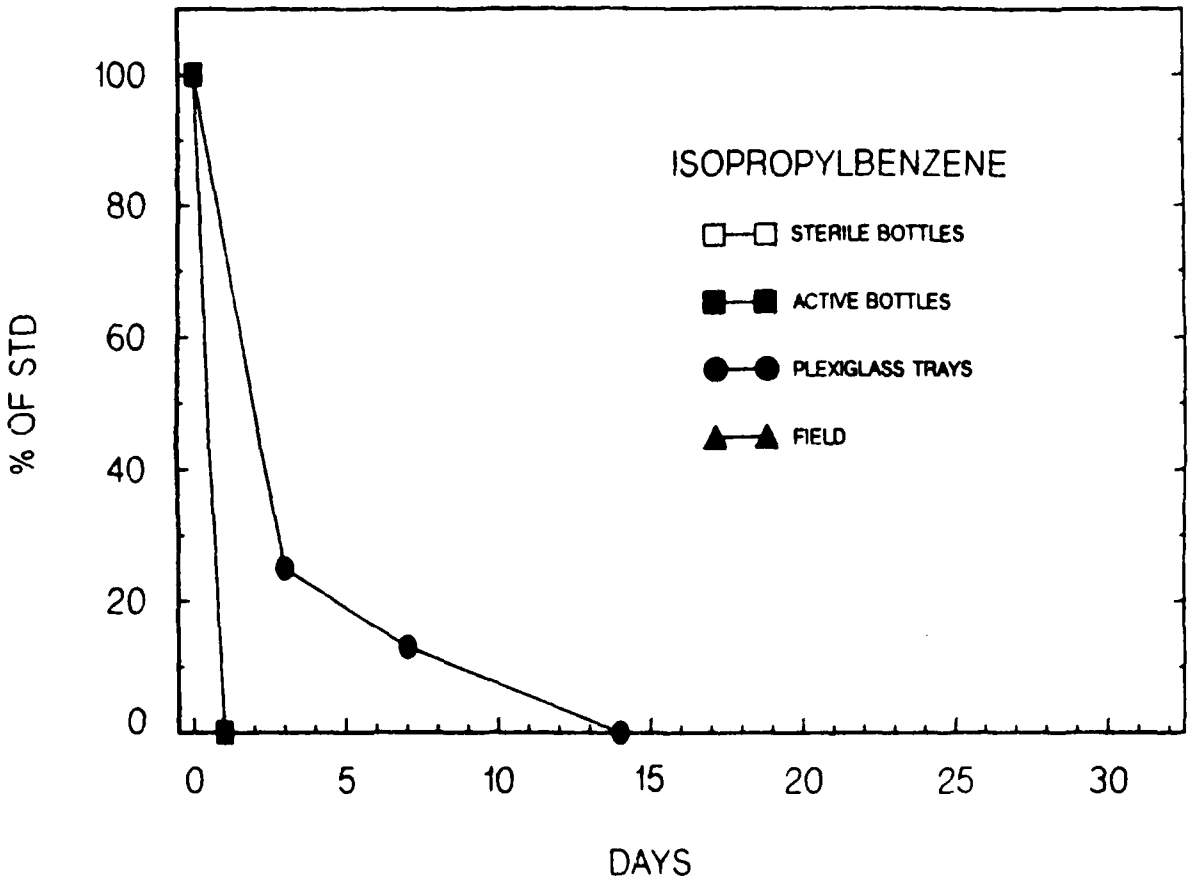



Figure 10. Change in Concentration Ratio (Expressed as Percent of Standard) of Isopropylbenzene to Tetradecane in Samples Taken from the Shallow Water Systems.

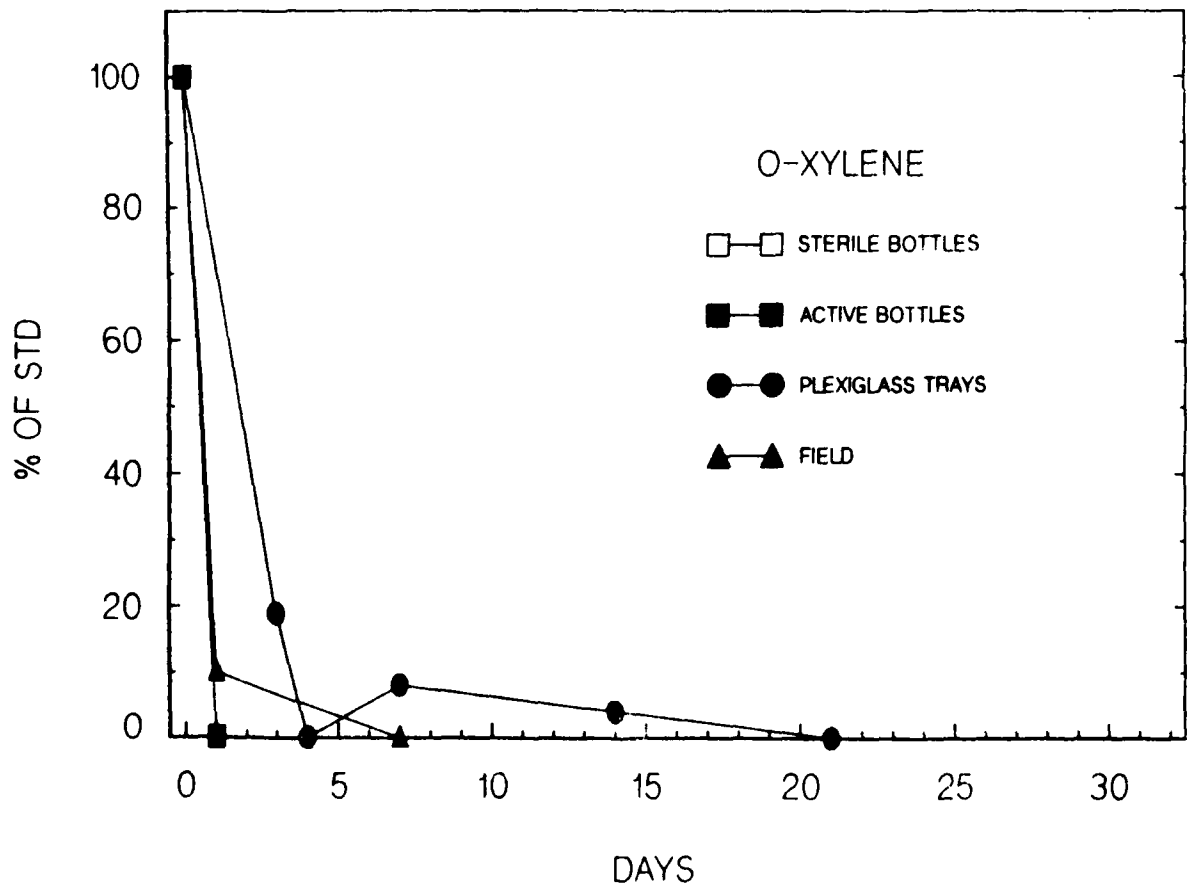



Figure 11. Change in Concentration Ratio (Expressed as Percent of Standard) of o-Xylene to Tetradecane in Samples Taken from the Shallow Water Systems.

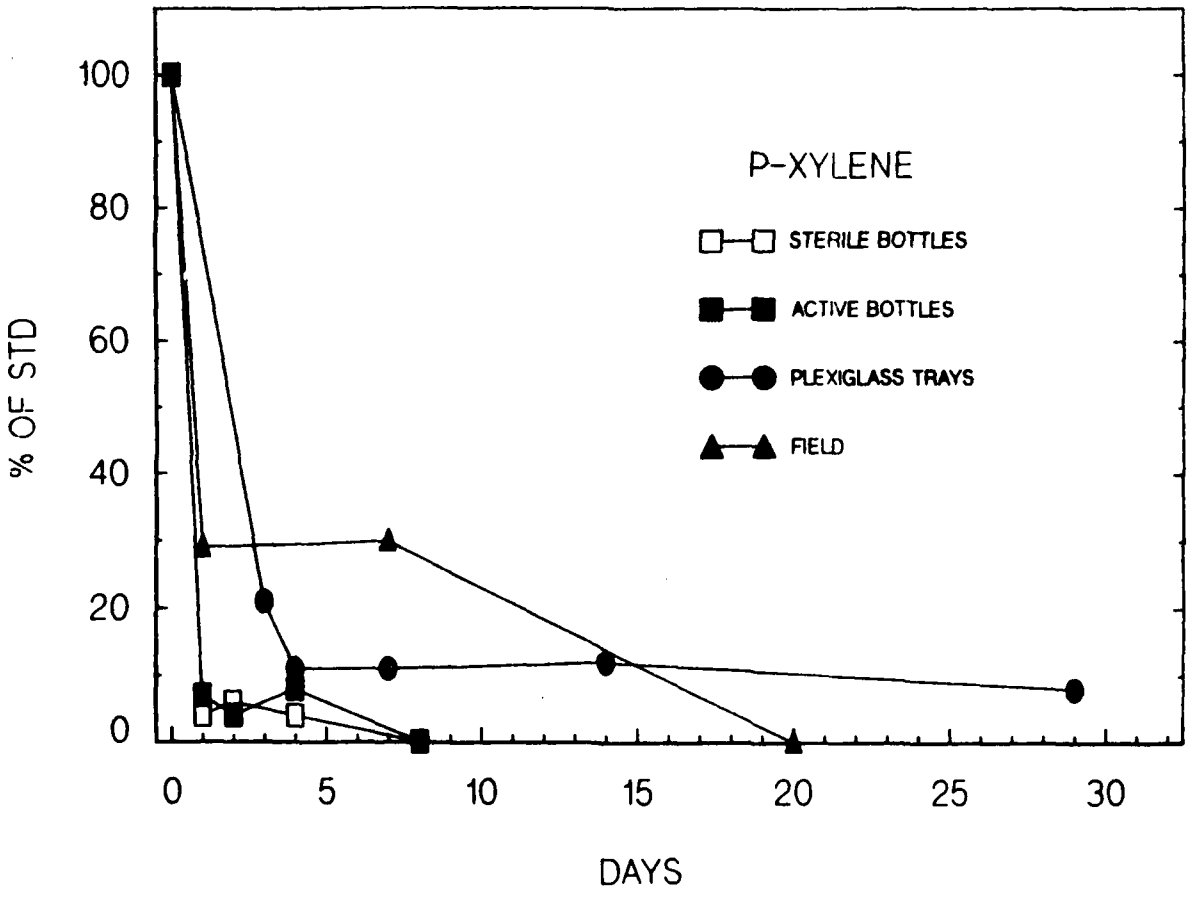



Figure 12. Change in Concentration Ratio (Expressed as Percent of Standard) of p-Xylene to Tetradecane in Samples Taken from the Shallow Water Systems.

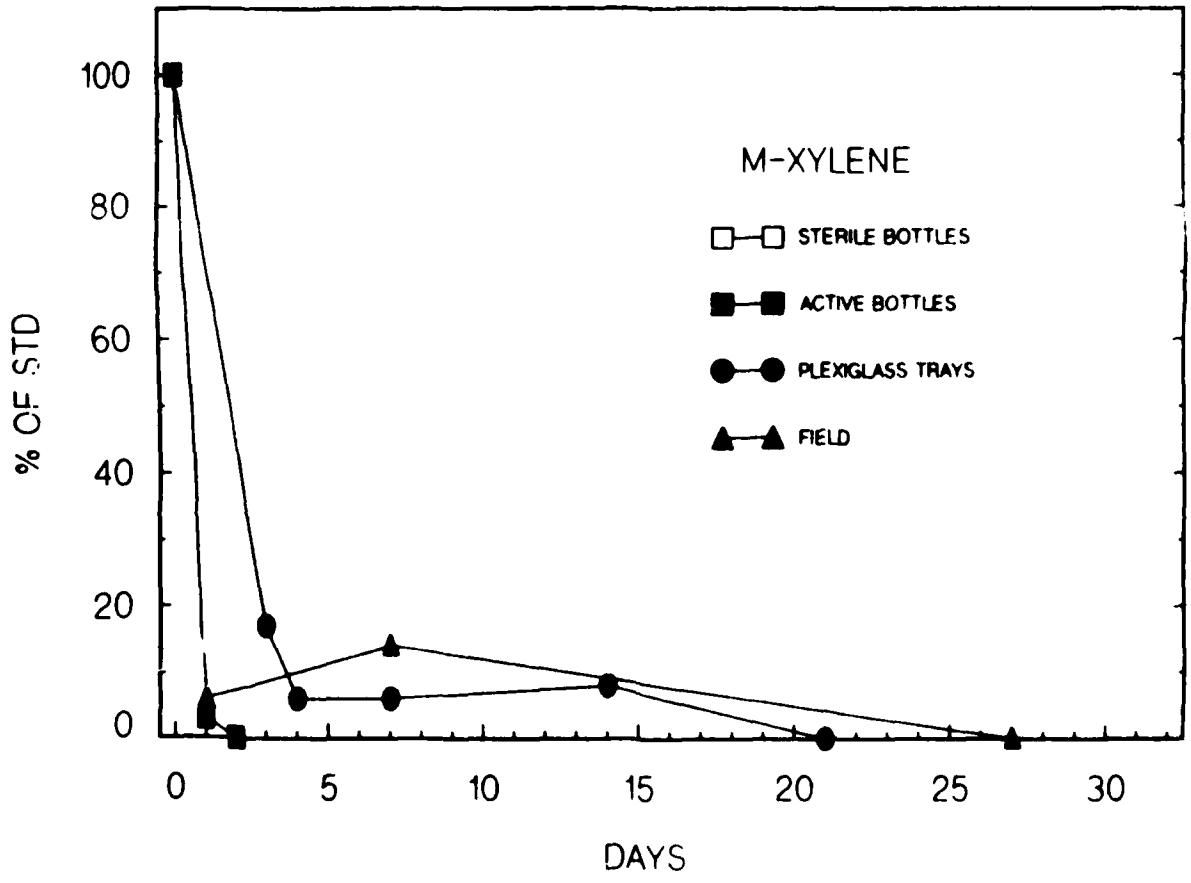



Figure 13. Change in Concentration Ratio (Expressed as Percent of Standard) of m-Xylene to Tetradecane in Samples Taken from the Shallow Water Systems.

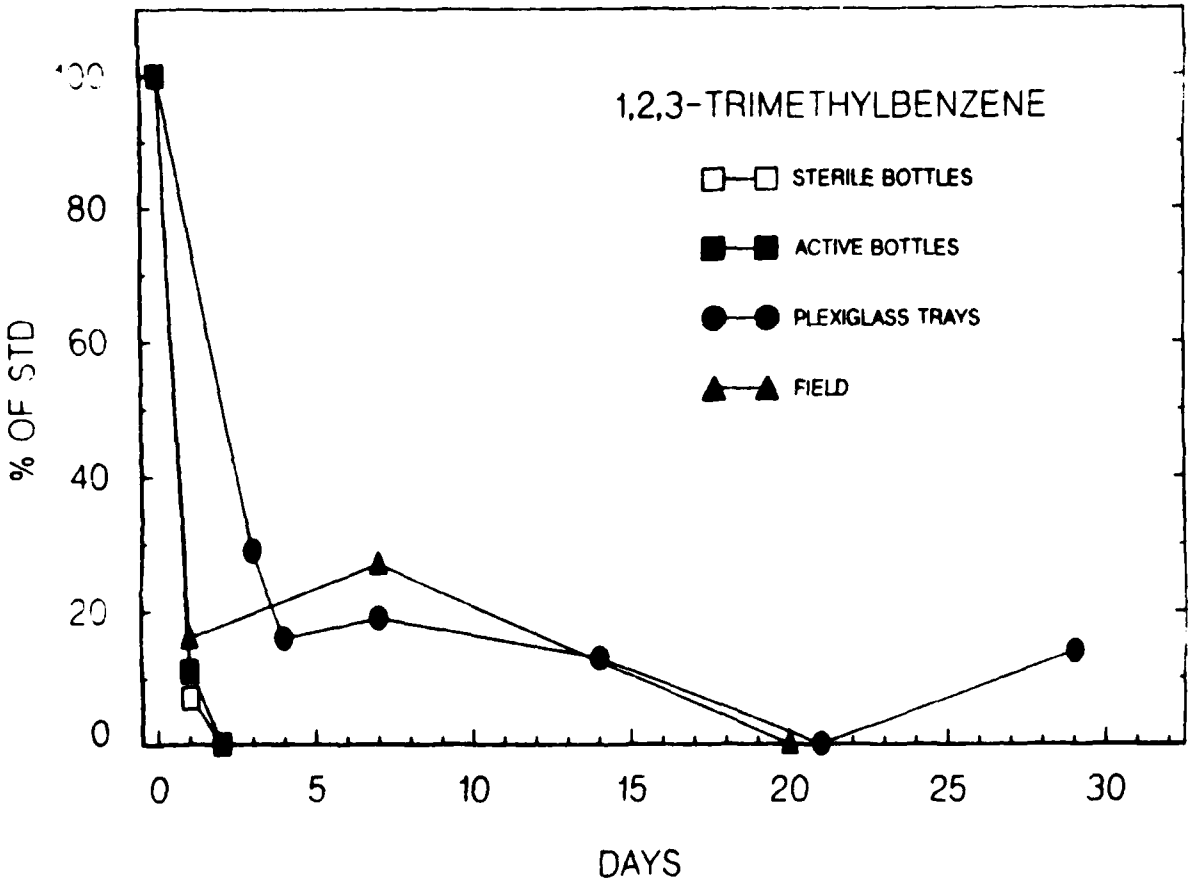



Figure 14. Change in Concentration Ratio (Expressed as Percent of Standard) of 1,2,3-Trimethylbenzene to Tetradecane in Samples Taken from the Shallow Water Systems.

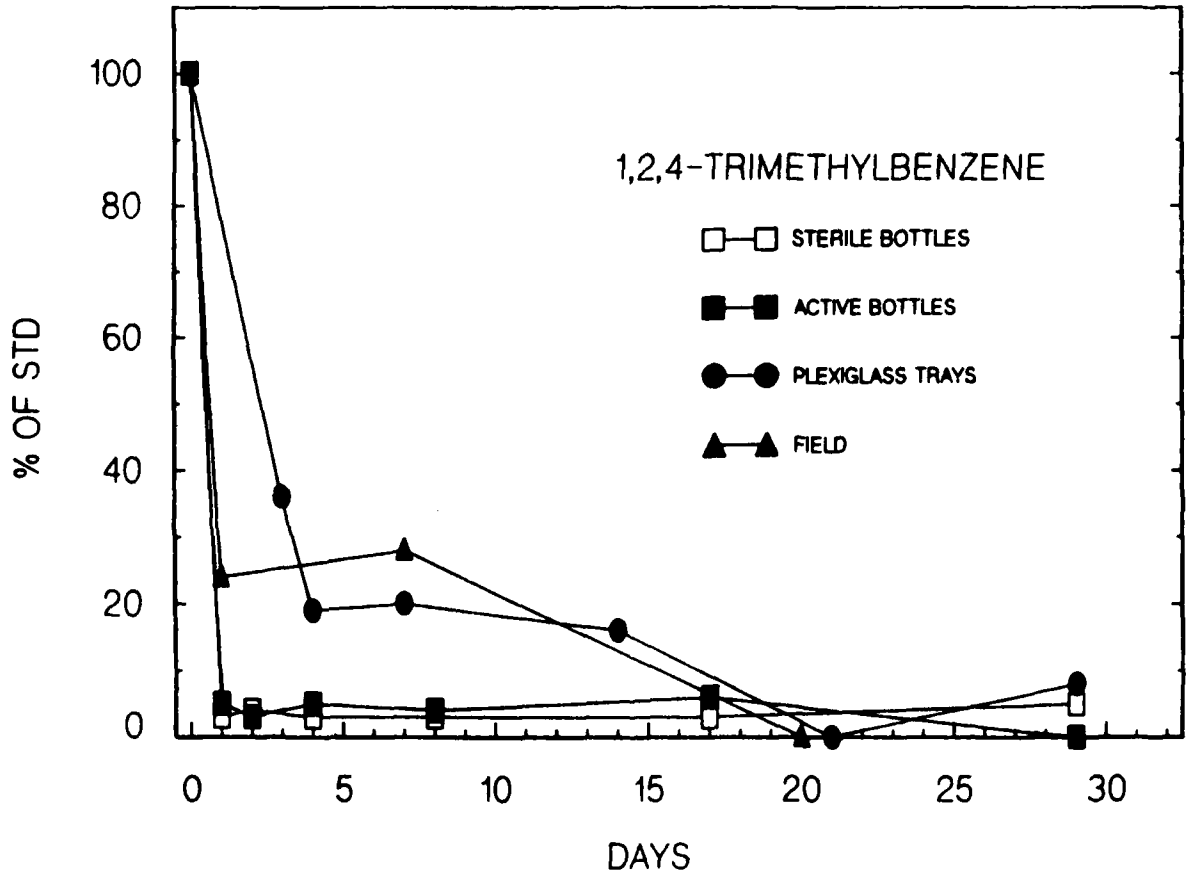



Figure 15. Change in Concentration Ratio (Expressed as Percent of Standard) of 1,2,4-Trimethylbenzene to Tetradecane in Samples Taken from the Shallow Water Systems.

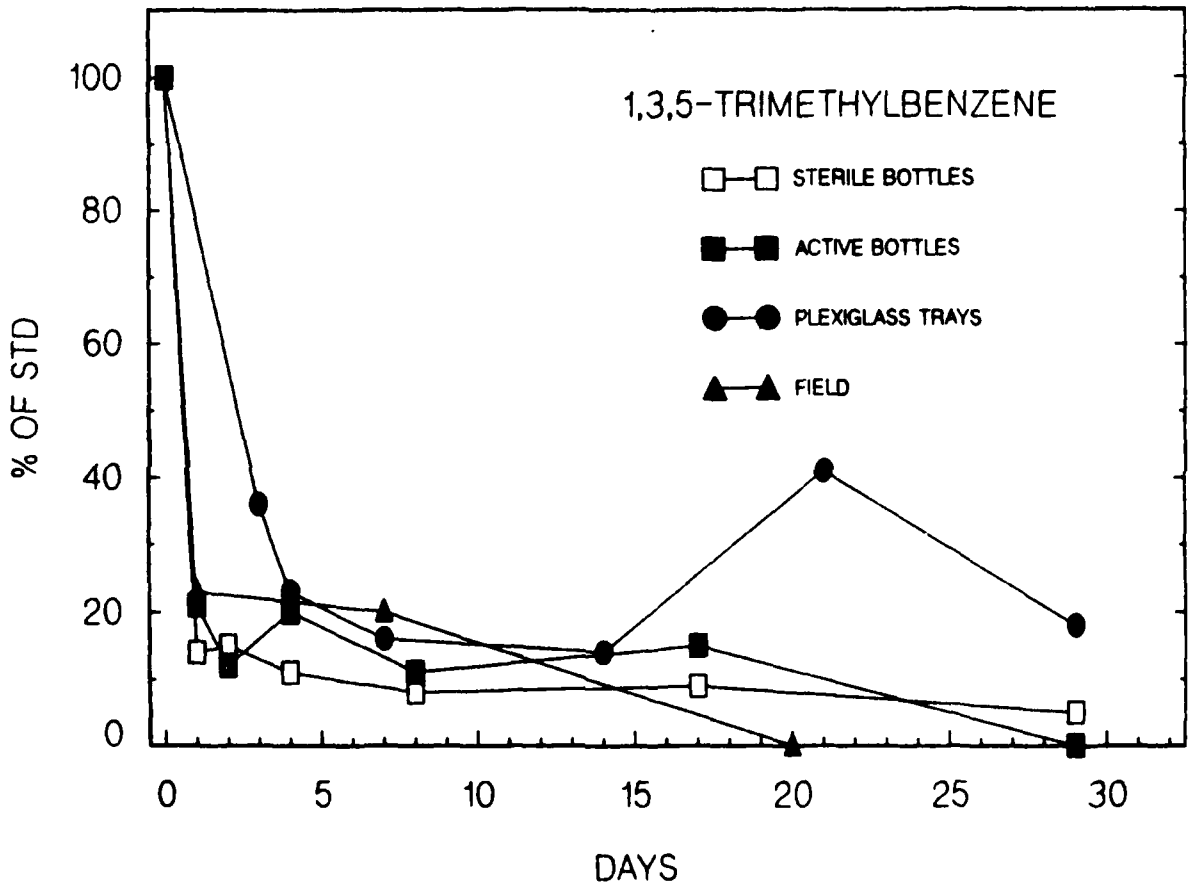



Figure 16. Change in Concentration Ratio (Expressed as Percent of Standard) of 1,3,5-Trimethylbenzene to Tetradecane in Samples Taken from the Shallow Water Systems.

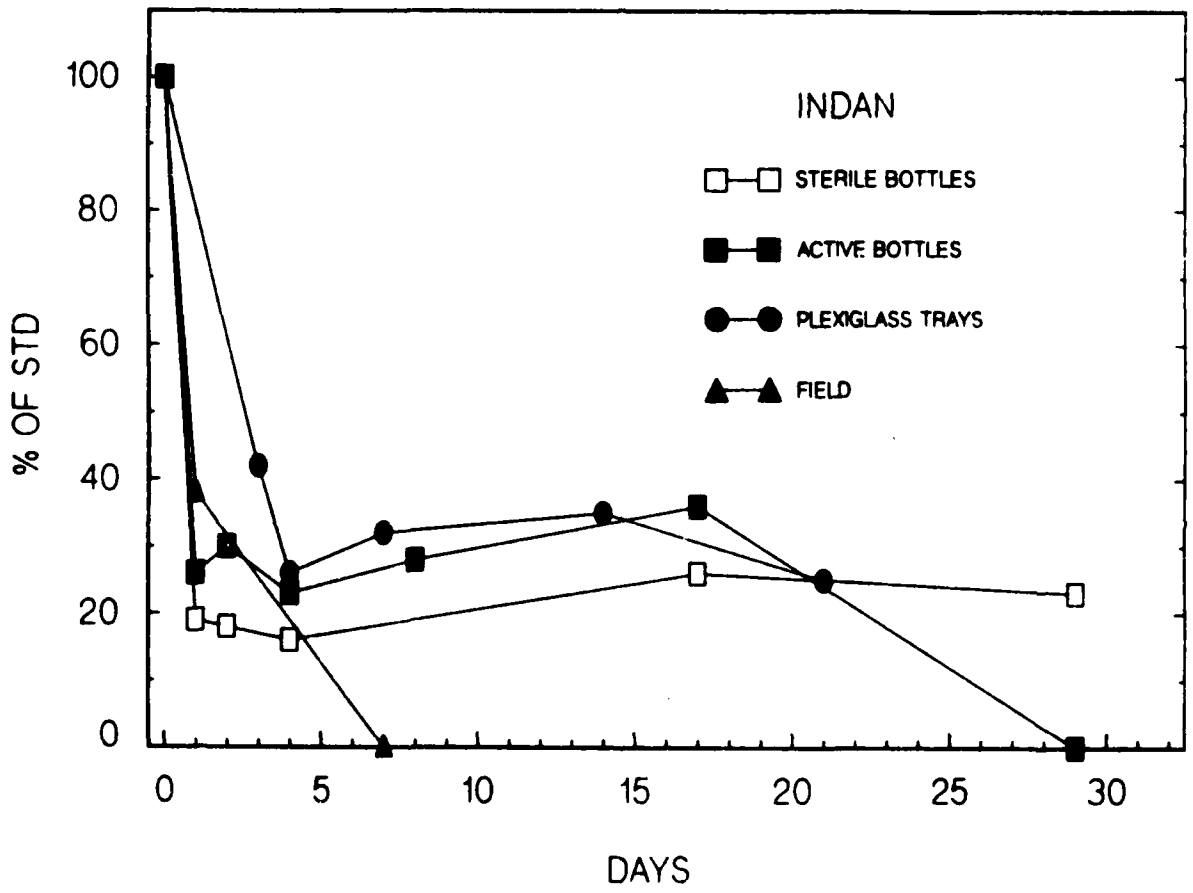



Figure 17. Change in Concentration Ratio (Expressed as Percent of Standard) of Indan to Tetradecane in Samples Taken from the Shallow Water Systems.

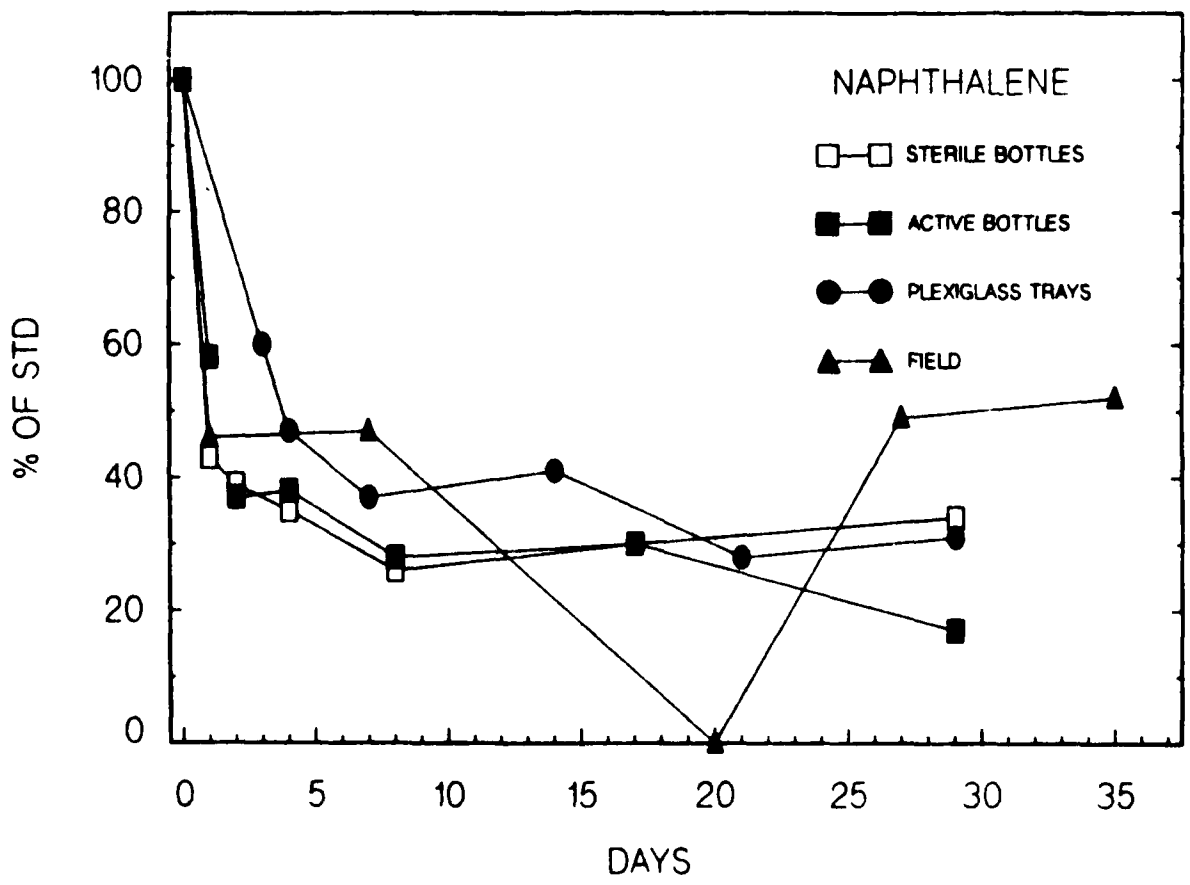



Figure 18. Change in Concentration Ratio (Expressed as Percent of Standard) of Naphthalene to Tetradecane in Samples Taken from the Shallow Water Systems.

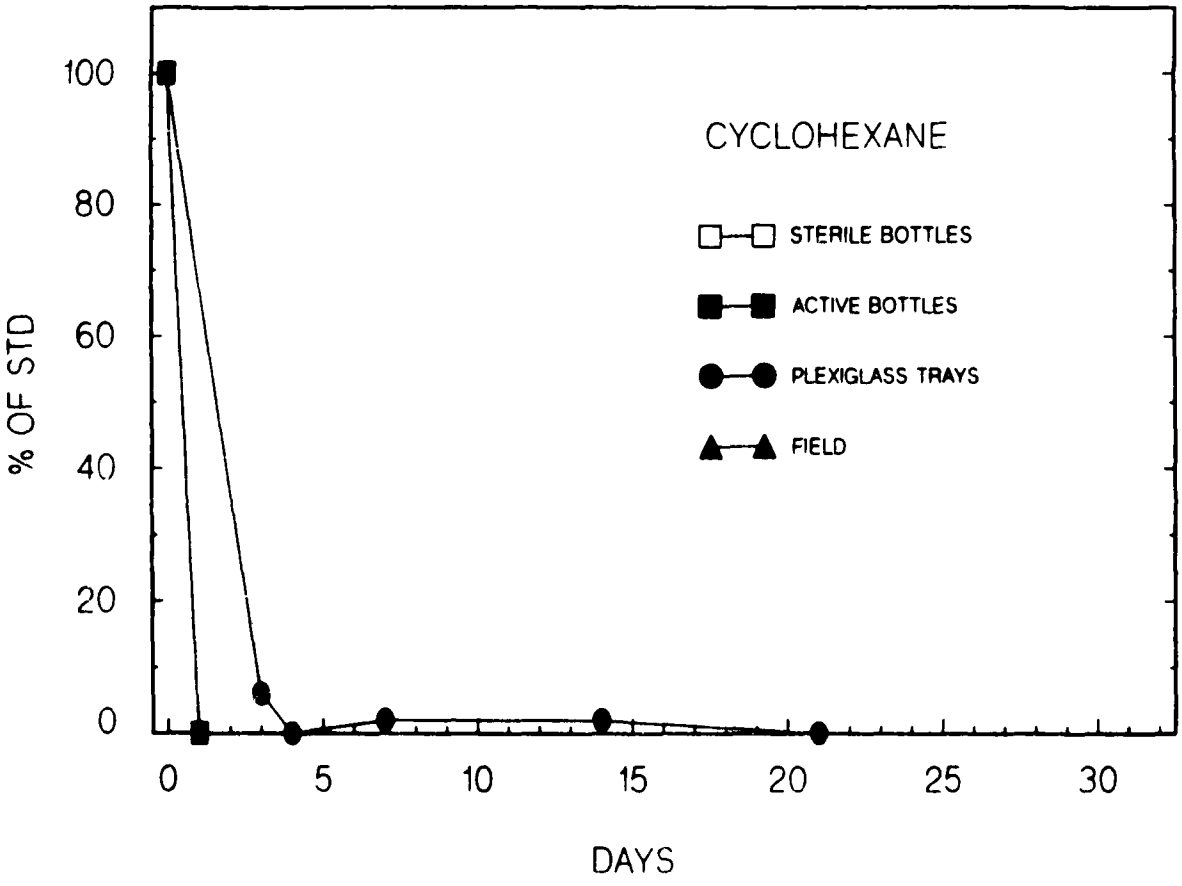



Figure 19. Change in Concentration Ratio (Expressed as Percent of Standard) of Cyclohexane to Tetradecane in Samples Taken from the Shallow Water Systems.

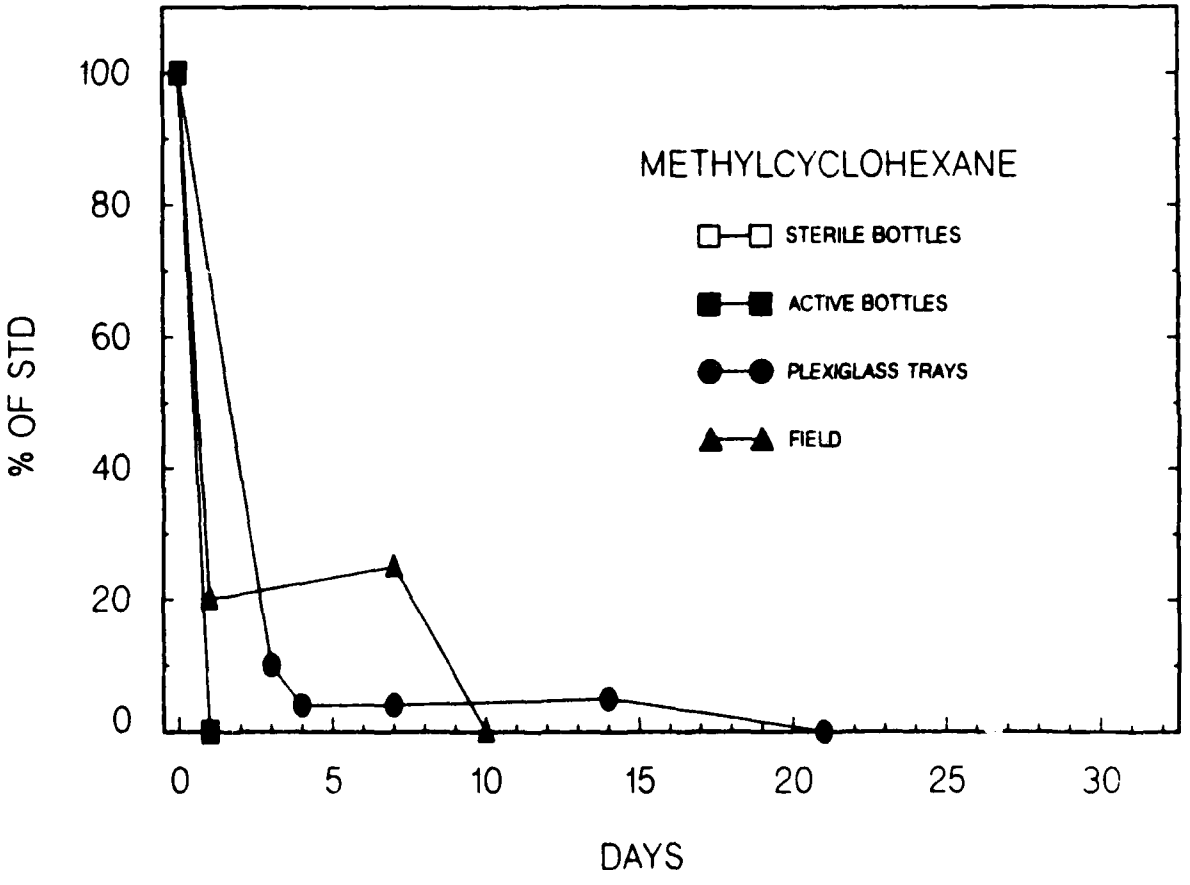



Figure 20. Change in Concentration Ratio (Expressed as Percent of Standard) of Methylcyclohexane to Tetradecane in Samples Taken from the Shallow Water Systems.

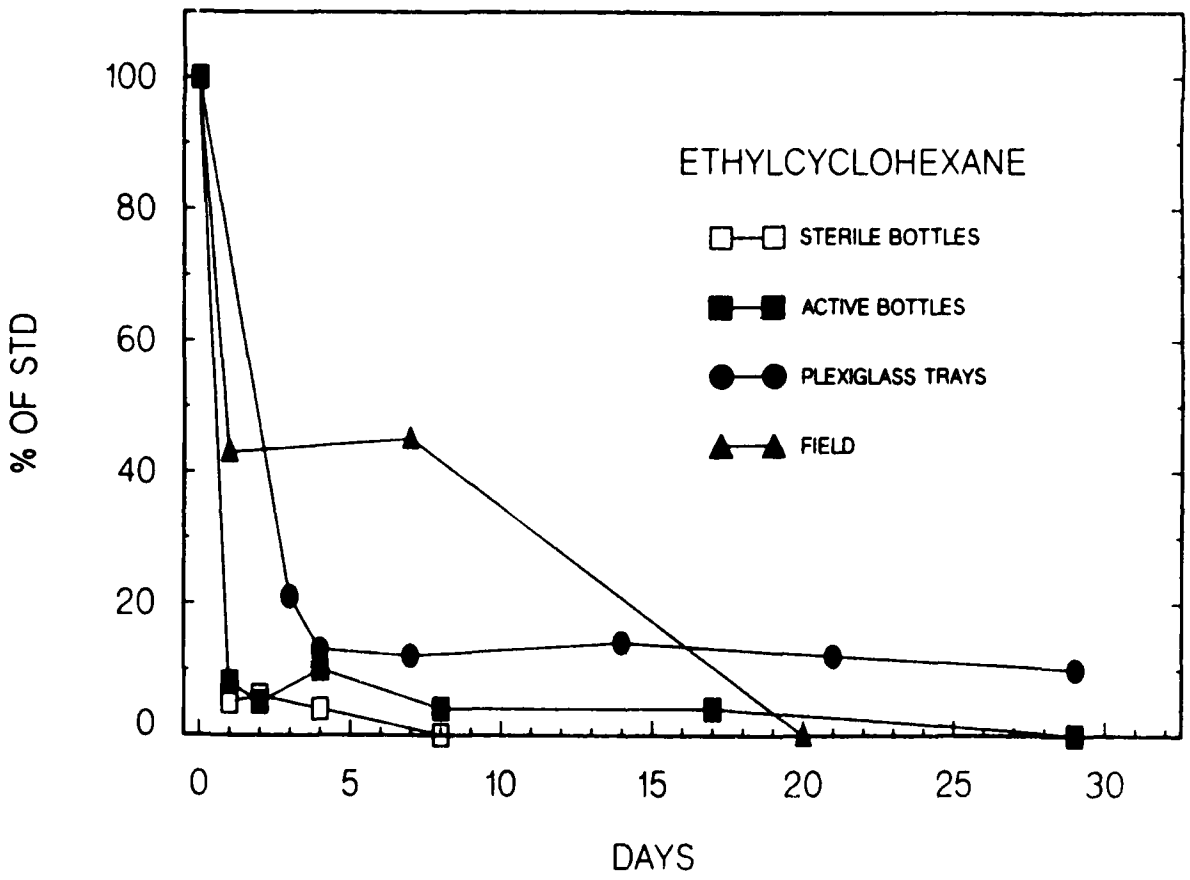



Figure 21. Change in Concentration Ratio (Expressed as Percent of Standard) of Ethylcyclohexane to Tetradecane in Samples Taken from the Shallow Water Systems.

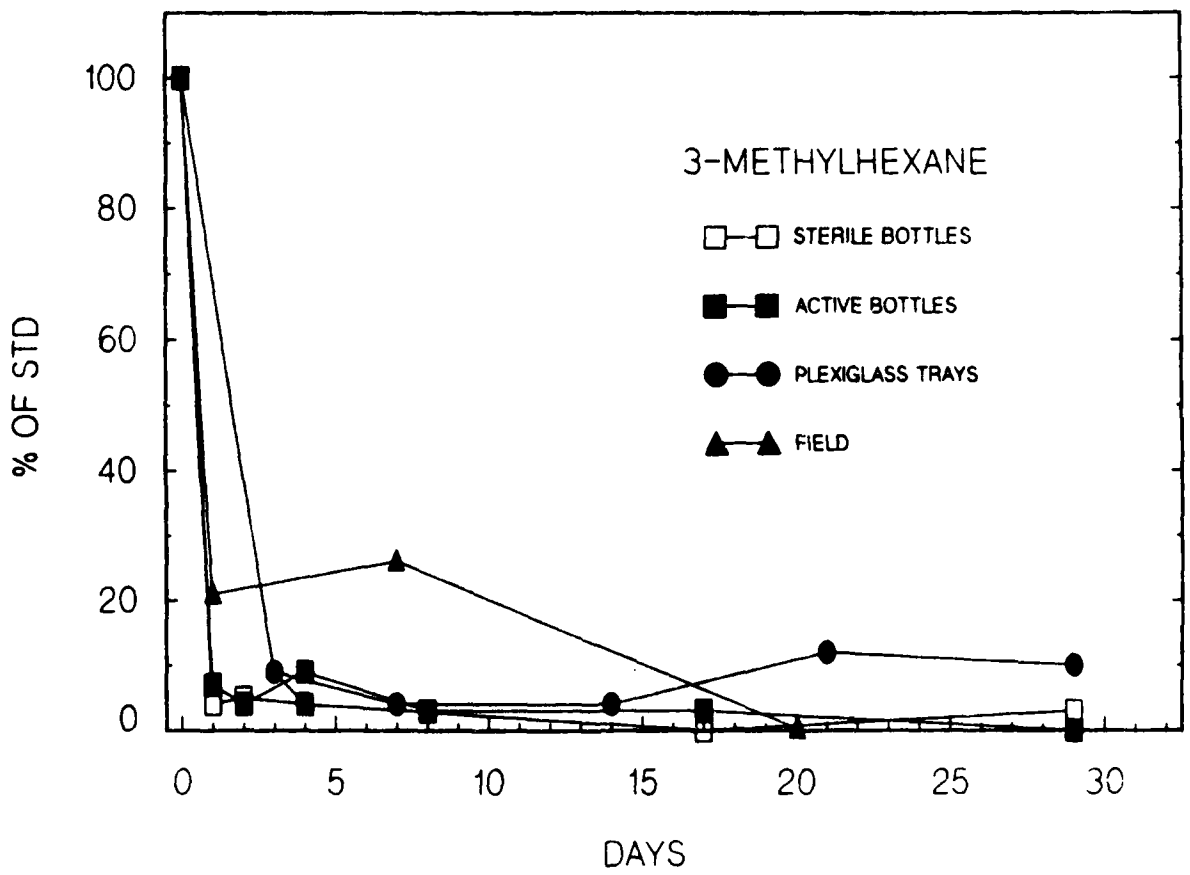



Figure 22. Change in Concentration Ratio (Expressed as Percent of Standard) of 3-Methylhexane to Tetradecane in Samples Taken from the Shallow Water Systems.

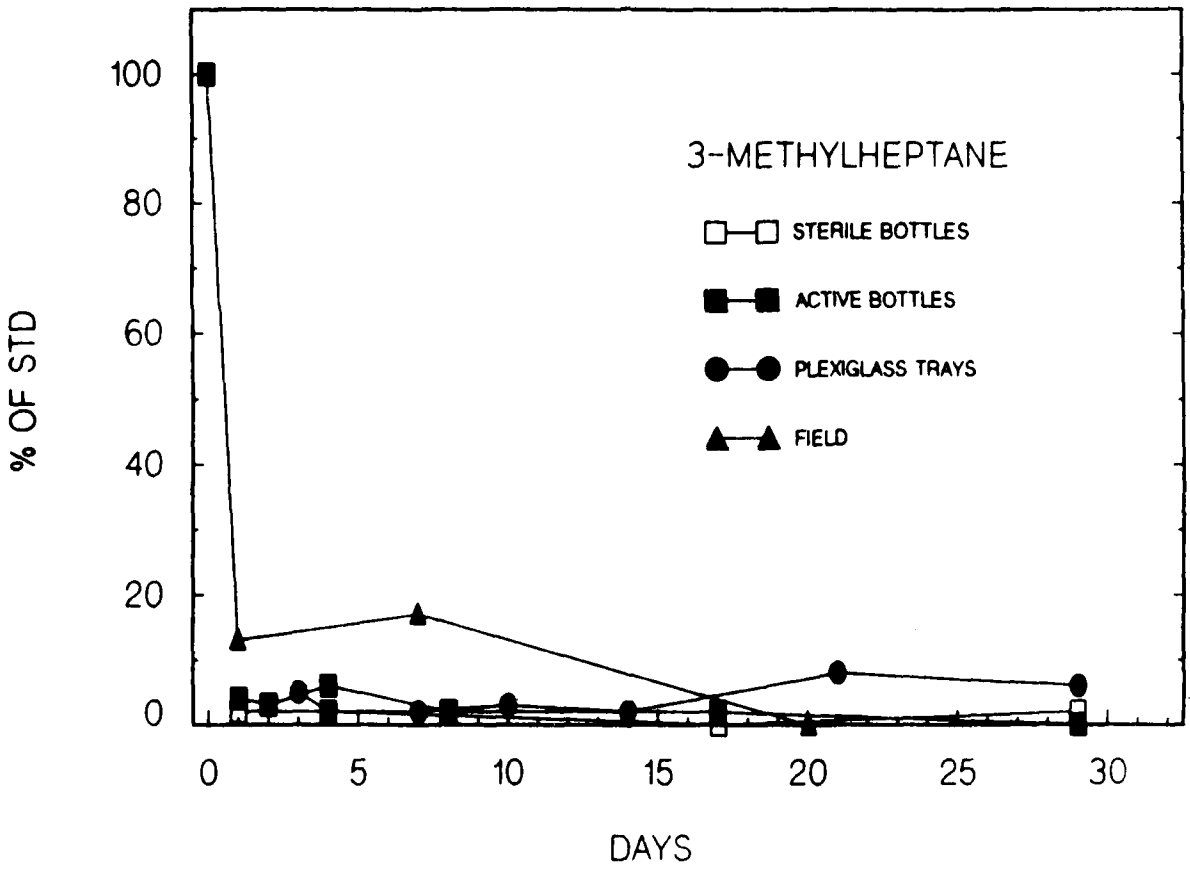



Figure 23. Change in Concentration Ratio (Expressed as Percent of Standard) of 3-Methylheptane to Tetradecane in Samples Taken from the Shallow Water Systems.

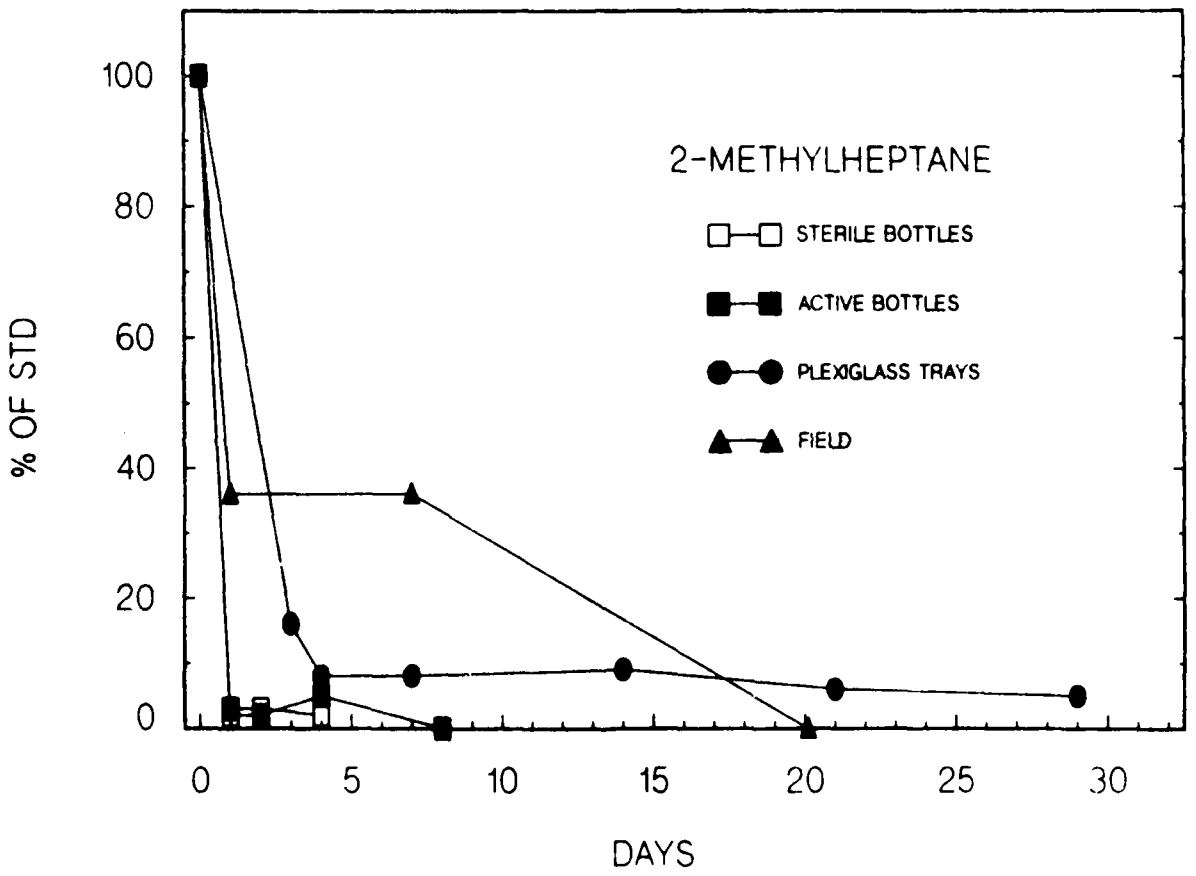



Figure 24. Change in Concentration Ratio (Expressed as Percent of Standard) of 2-Methylheptane to Tetradecane in Samples Taken from the Shallow Water Systems.

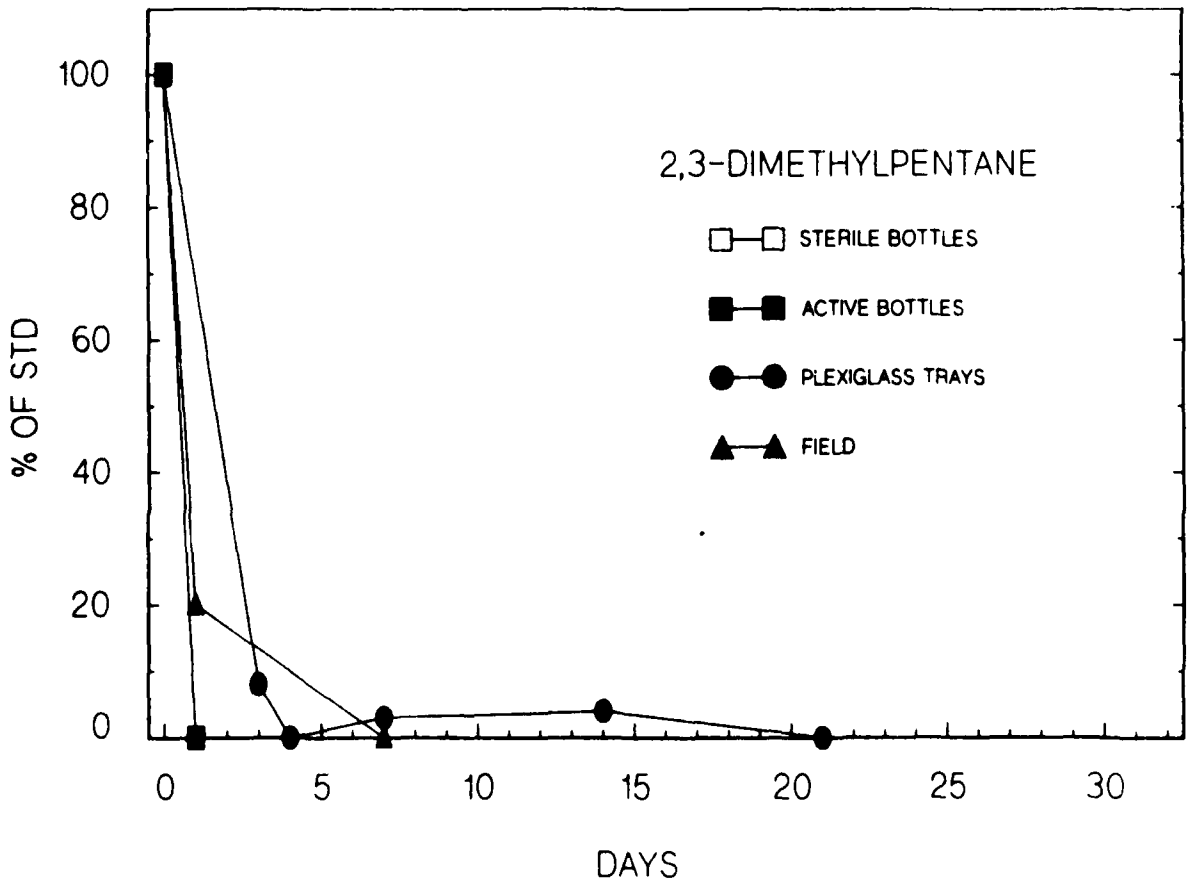



Figure 25. Change in Concentration Ratio (Expressed as Percent of Standard) of 2,3-Dimethylheptane to Tetradecane in Samples Taken from the Shallow Water Systems.

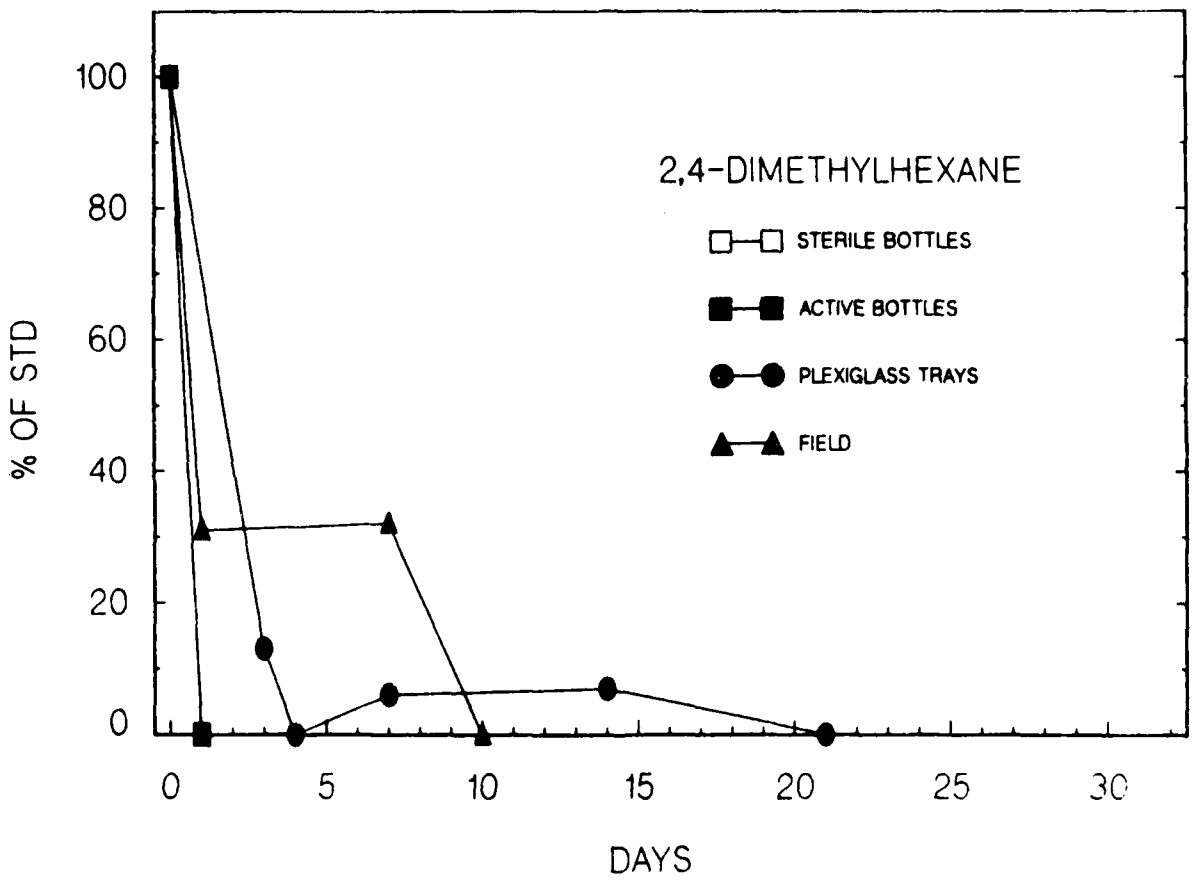



Figure 26. Change in Concentration Ratio (Expressed as Percent of Standard) of 2,4-Dimethylhexane to Tetradecane in Samples Taken from the Shallow Water Systems.

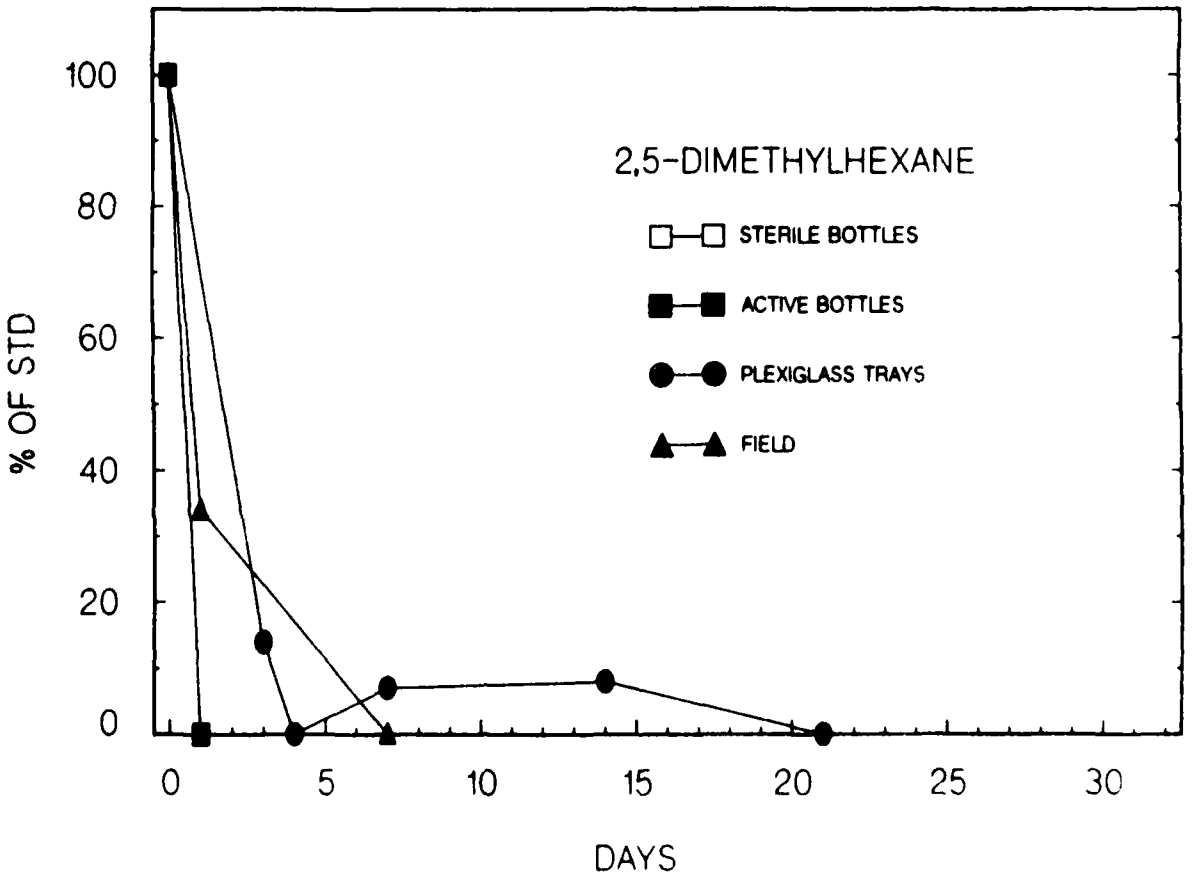



Figure 27. Change in Concentration Ratio (Expressed as Percent of Standard) of 2,5-Dimethylhexane to Tetradecane in Samples Taken from the Shallow Water Systems.

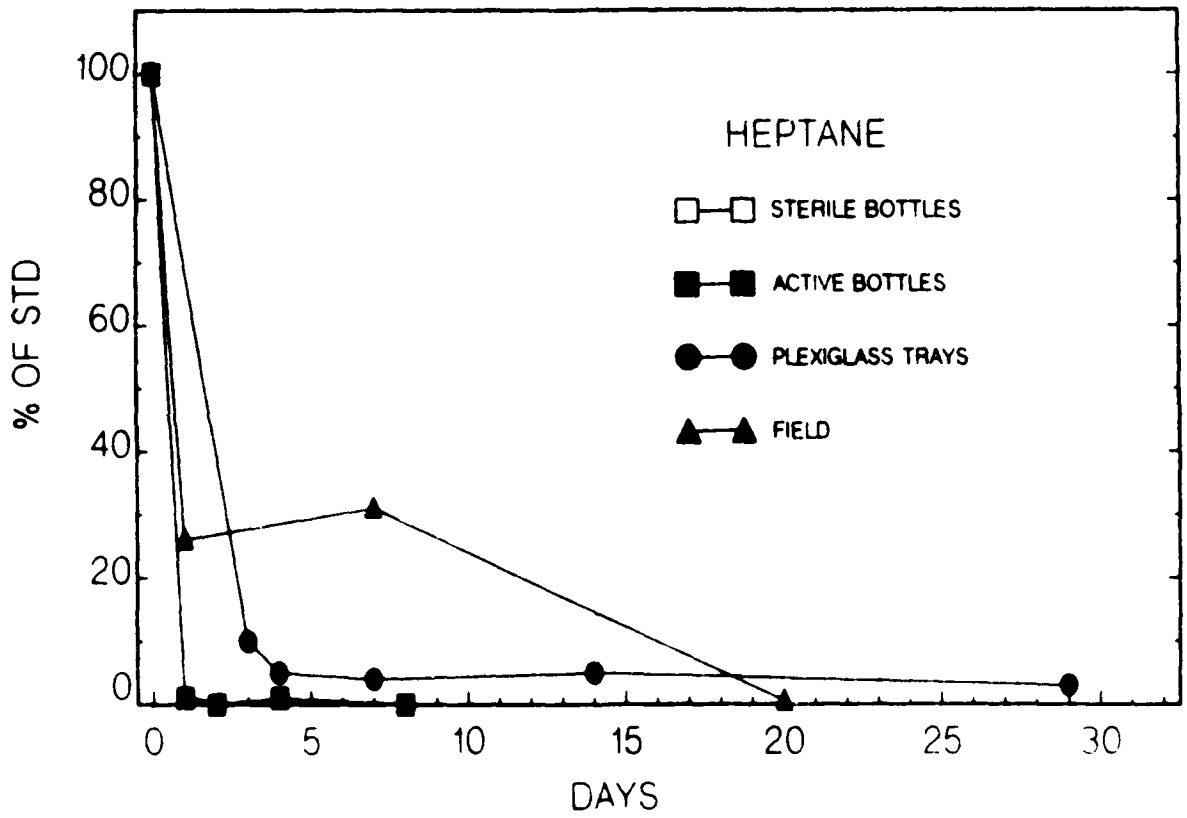



Figure 28. Change in Concentration Ratio (Expressed as Percent of Standard) of Heptane to Tetradecane in Samples Taken from the Shallow Water Systems.

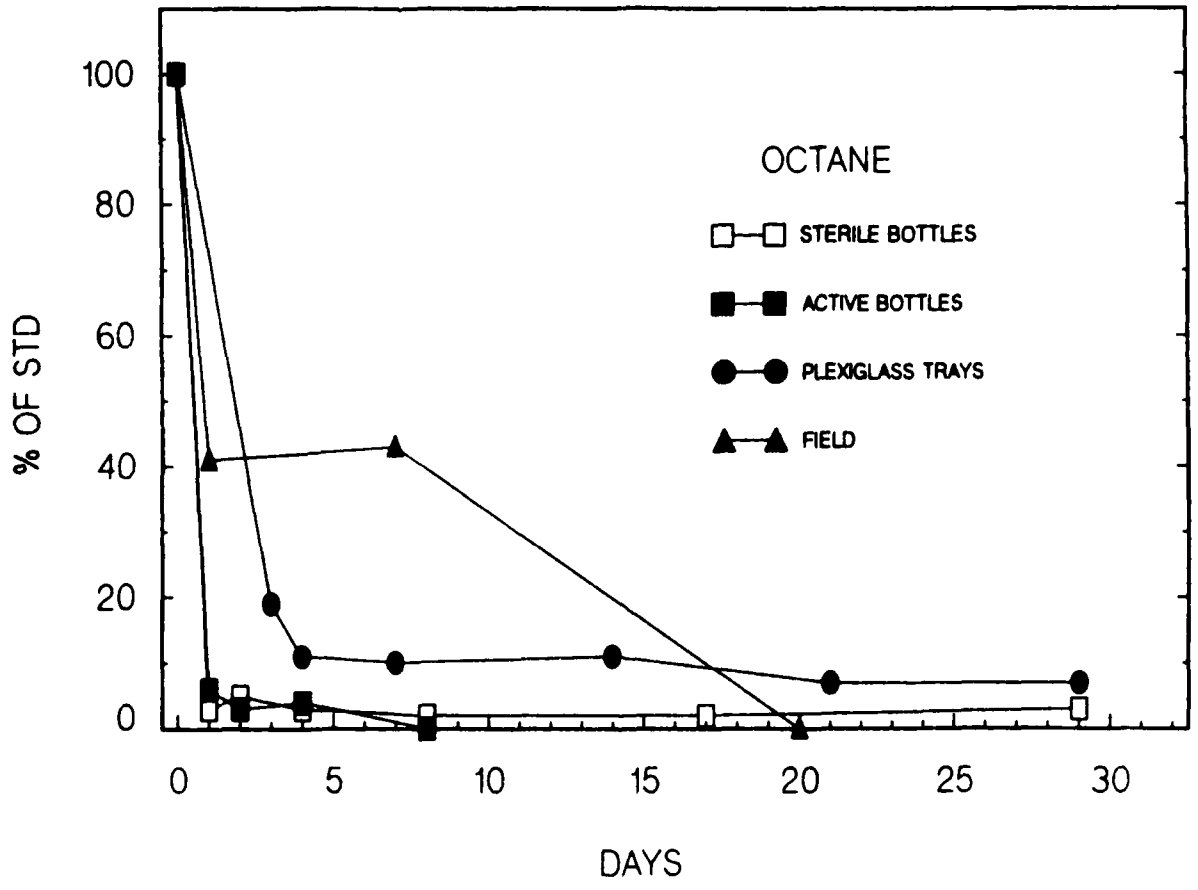



Figure 29. Change in Concentration Ratio (Expressed as Percent of Standard) of Octane to Tetradecane in Samples Taken from the Shallow Water Systems.

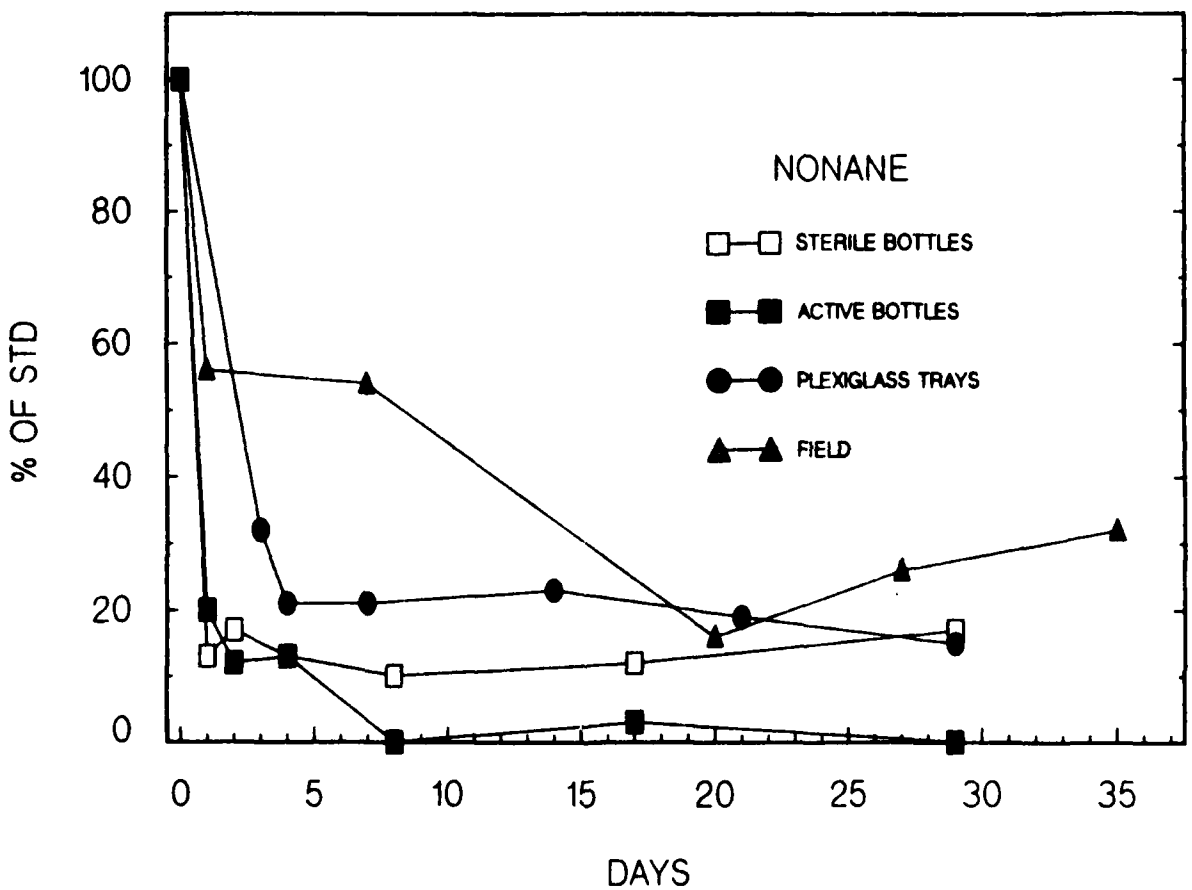



Figure 30. Change in Concentration Ratio (Expressed as Percent of Standard) of Nonane to Tetradecane in Samples Taken from the Shallow Water Systems.

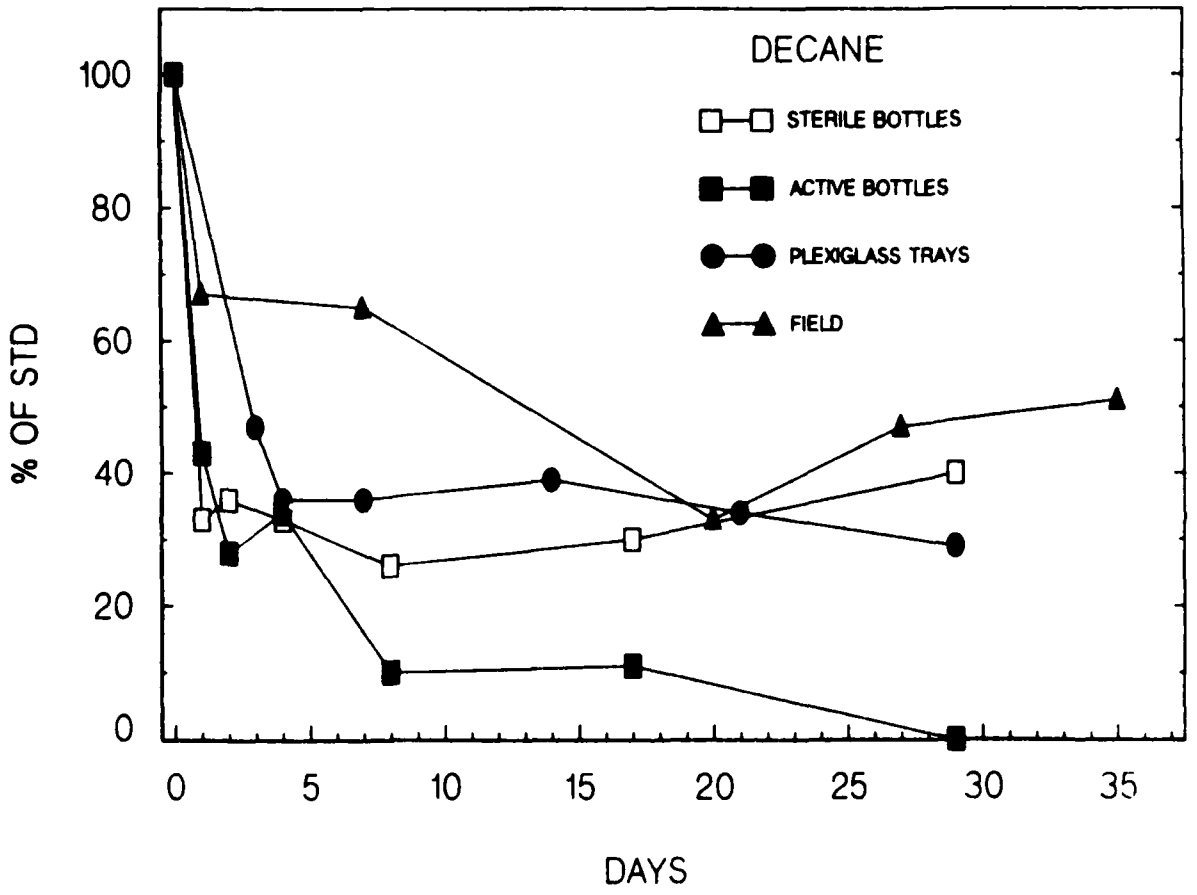



Figure 31. Change in Concentration Ratio (Expressed as Percent of Standard) of Decane to Tetradecane in Samples Taken from the Shallow Water Systems.

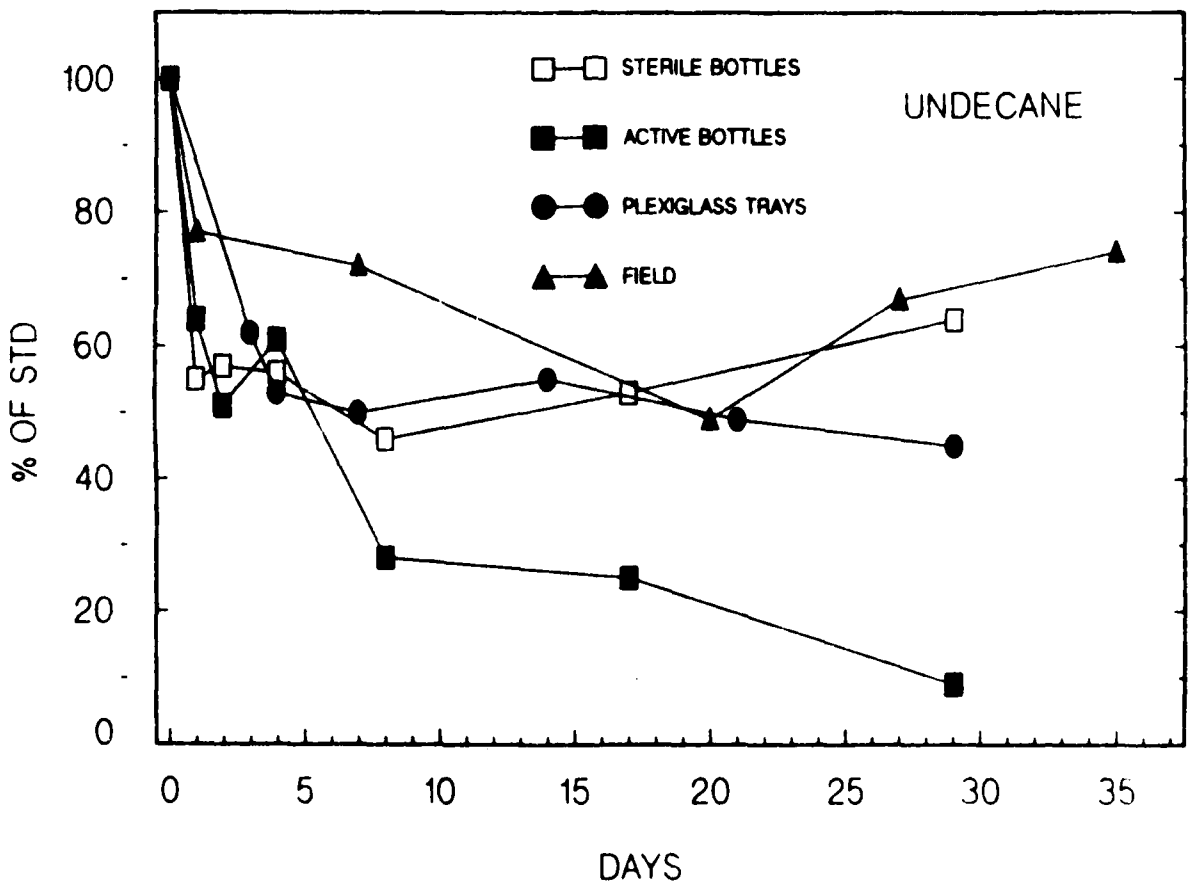



Figure 32. Change in Concentration Ratio (Expressed as Percent of Standard) of Undecane to Tetradecane in Samples Taken from the Shallow Water Systems.

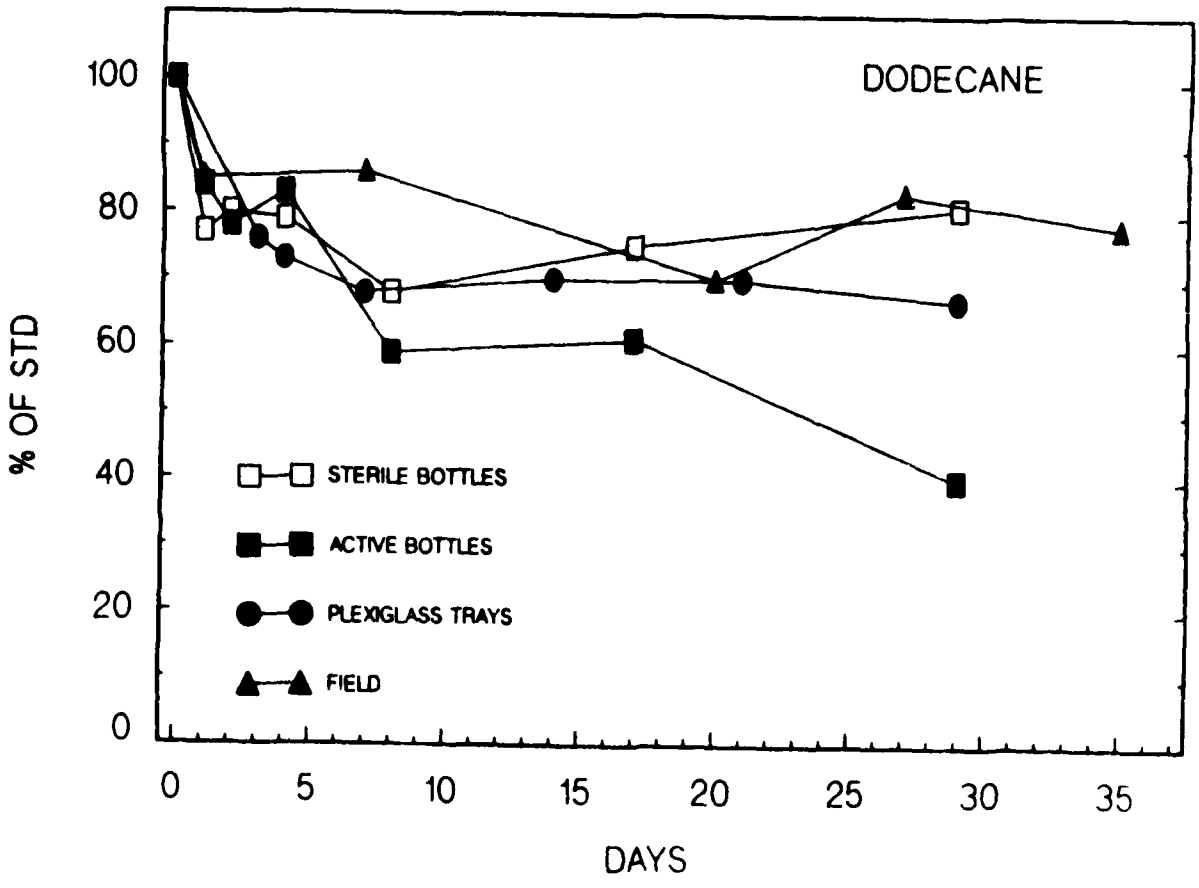



Figure 33. Change in Concentration Ratio (Expressed as Percent of Standard) of Dodecane to Tetradecane in Samples Taken from the Shallow Water Systems.

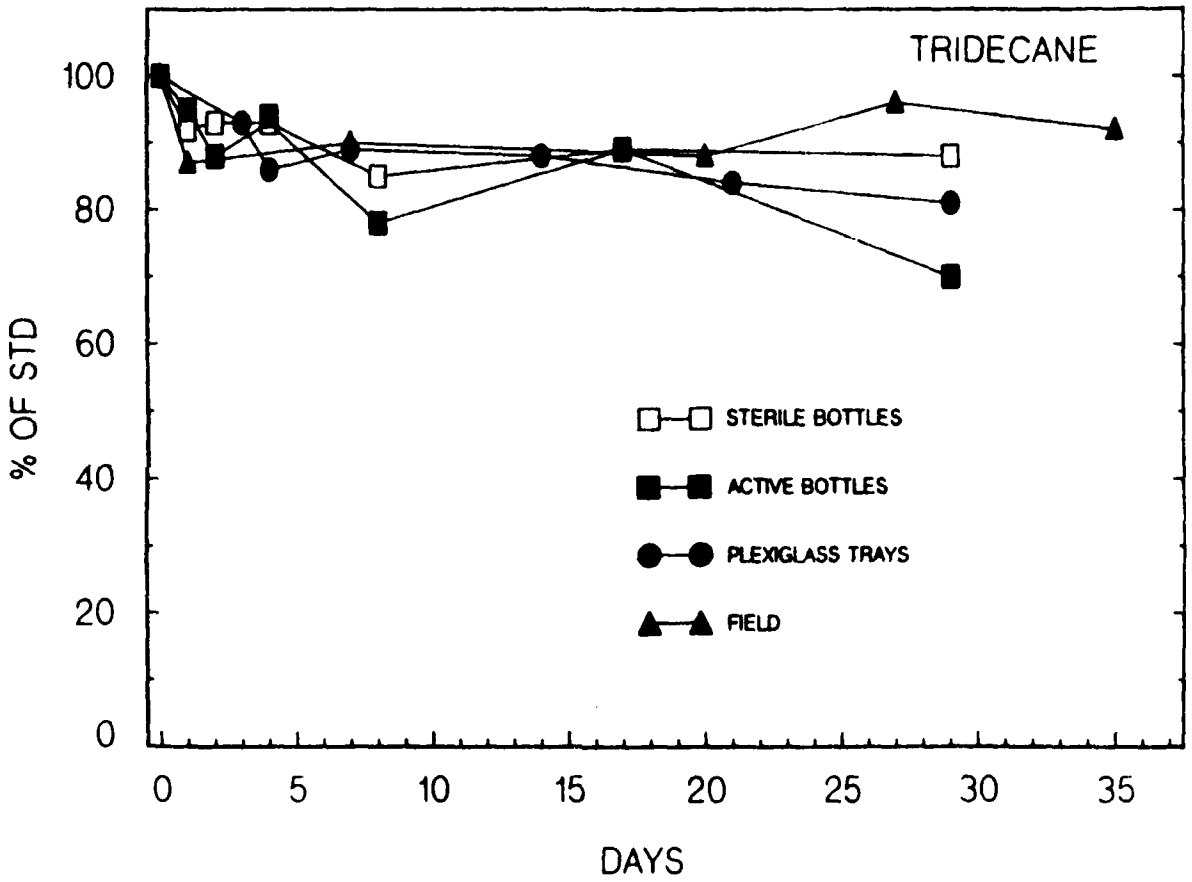



Figure 34. Change in Concentration Ratio (Expressed as Percent of Standard) of Tridecane to Tetradecane in Samples Taken from the Shallow Water Systems.

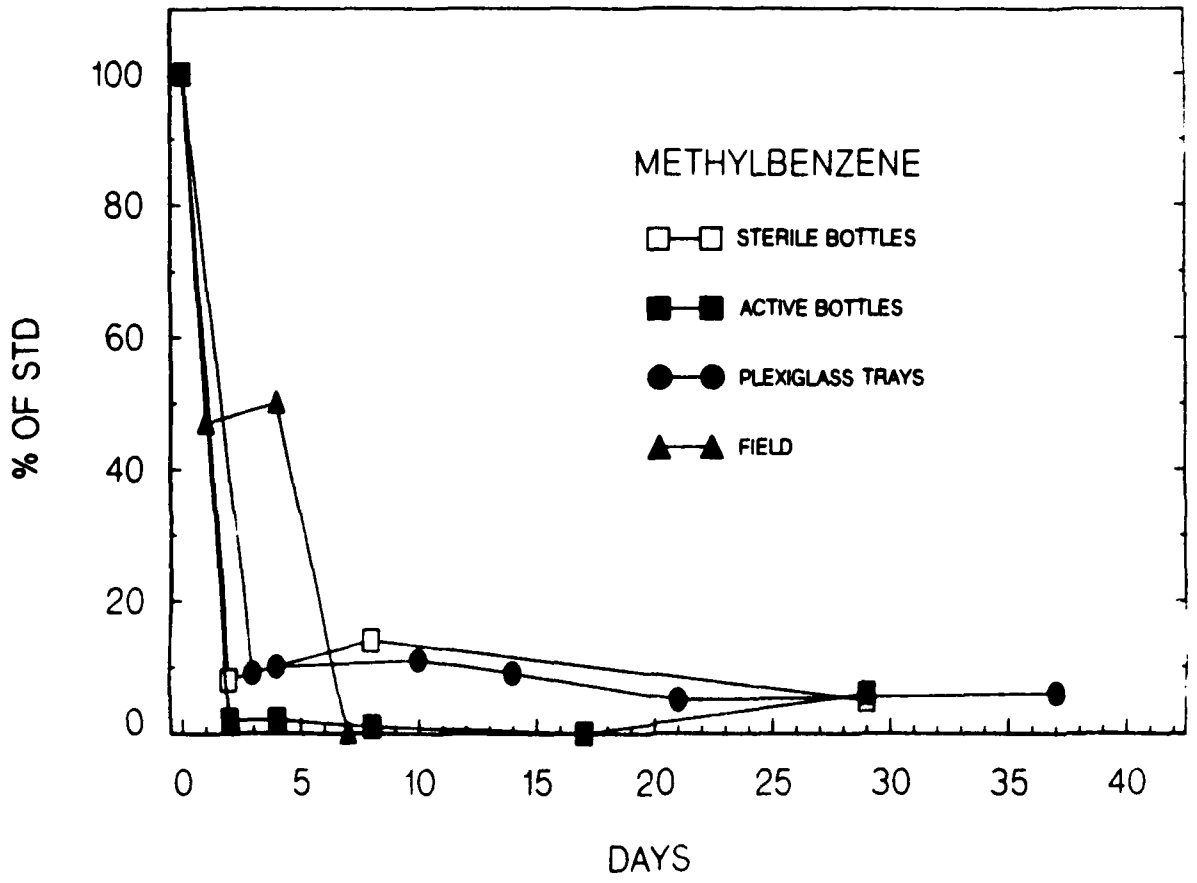



Figure 35. Change in Concentration Ratio (Expressed as Percent of Standard) of Methylbenzene to Tetradecane in Samples Taken from the Deep Water Systems.

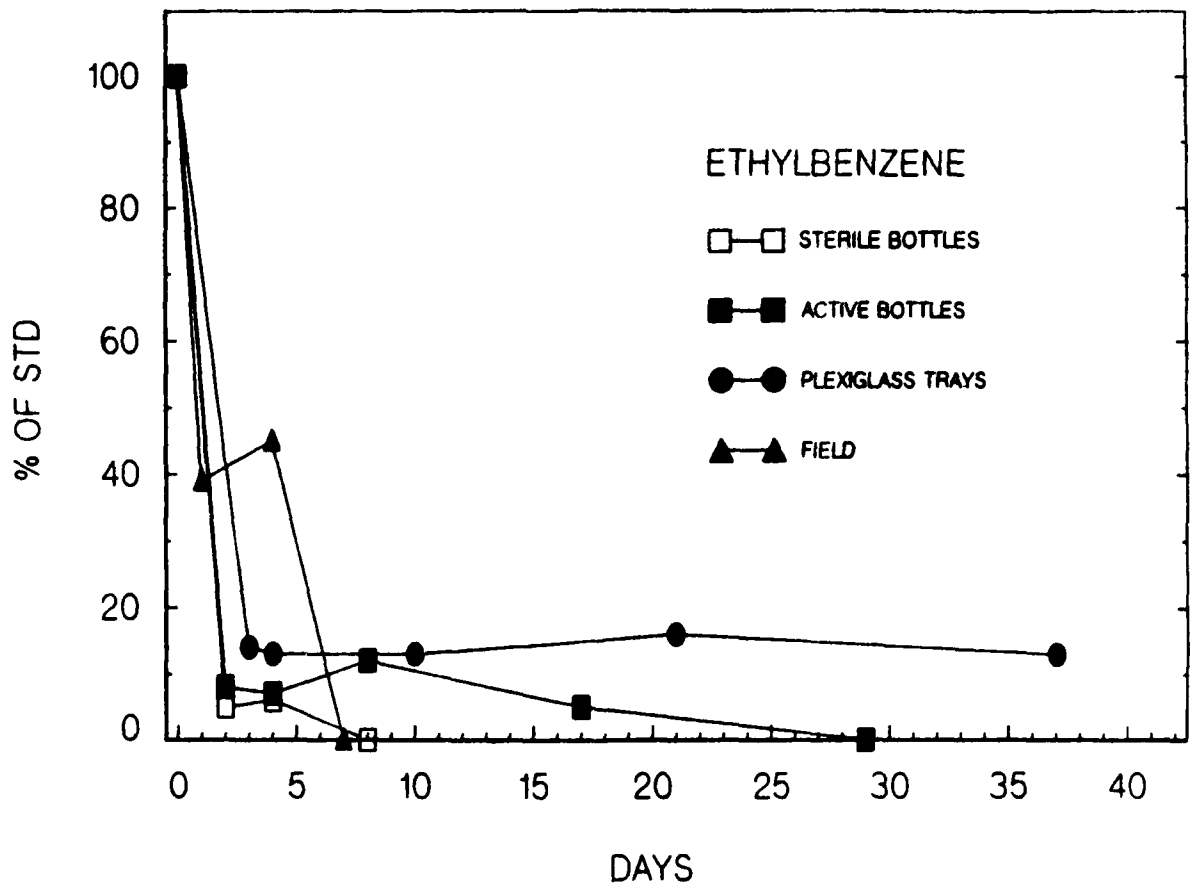



Figure 36. Change in Concentration Ratio (Expressed as Percent of Standard) of Ethylbenzene to Tetradecane in Samples Taken from the Deep Water Systems.

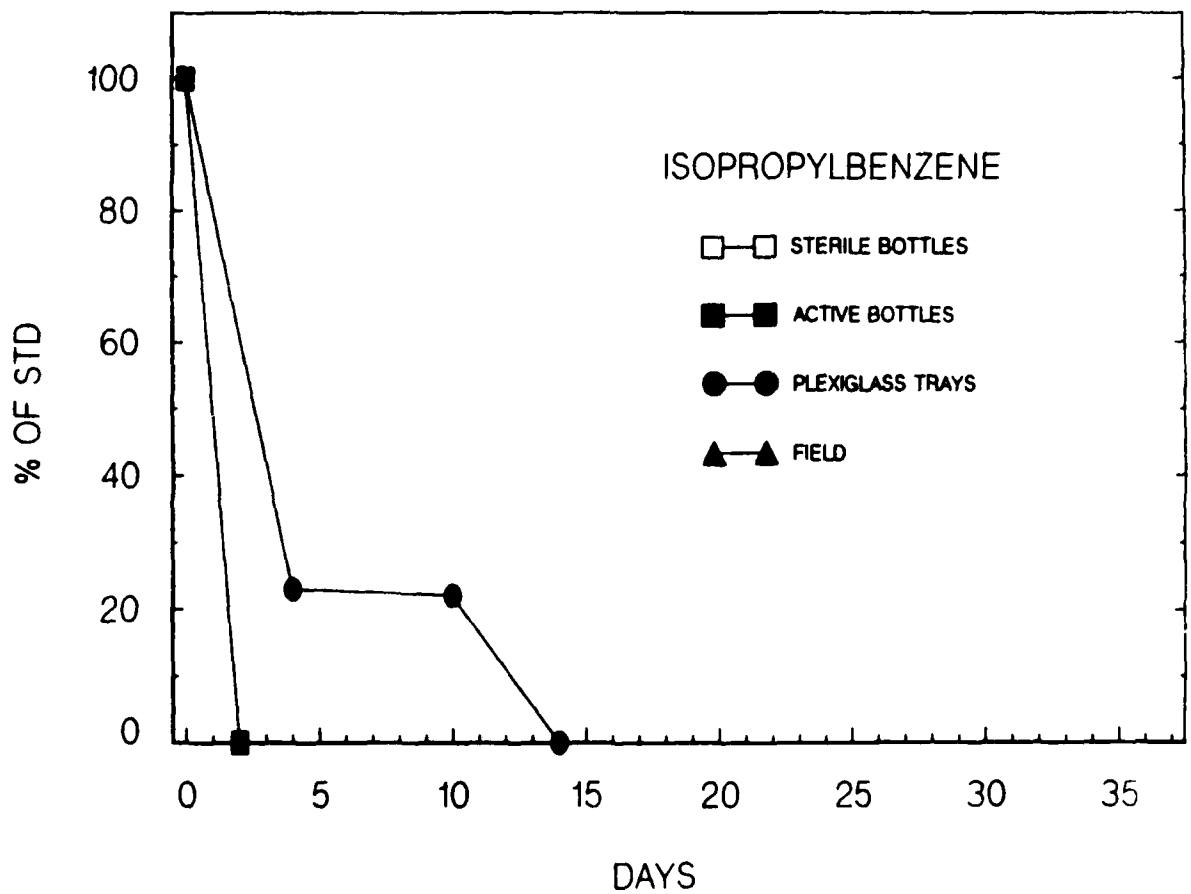



Figure 37. Change in Concentration Ratio (Expressed as Percent of Standard) of Isopropylbenzene to Tetradecane in Samples Taken from the Deep Water Systems.

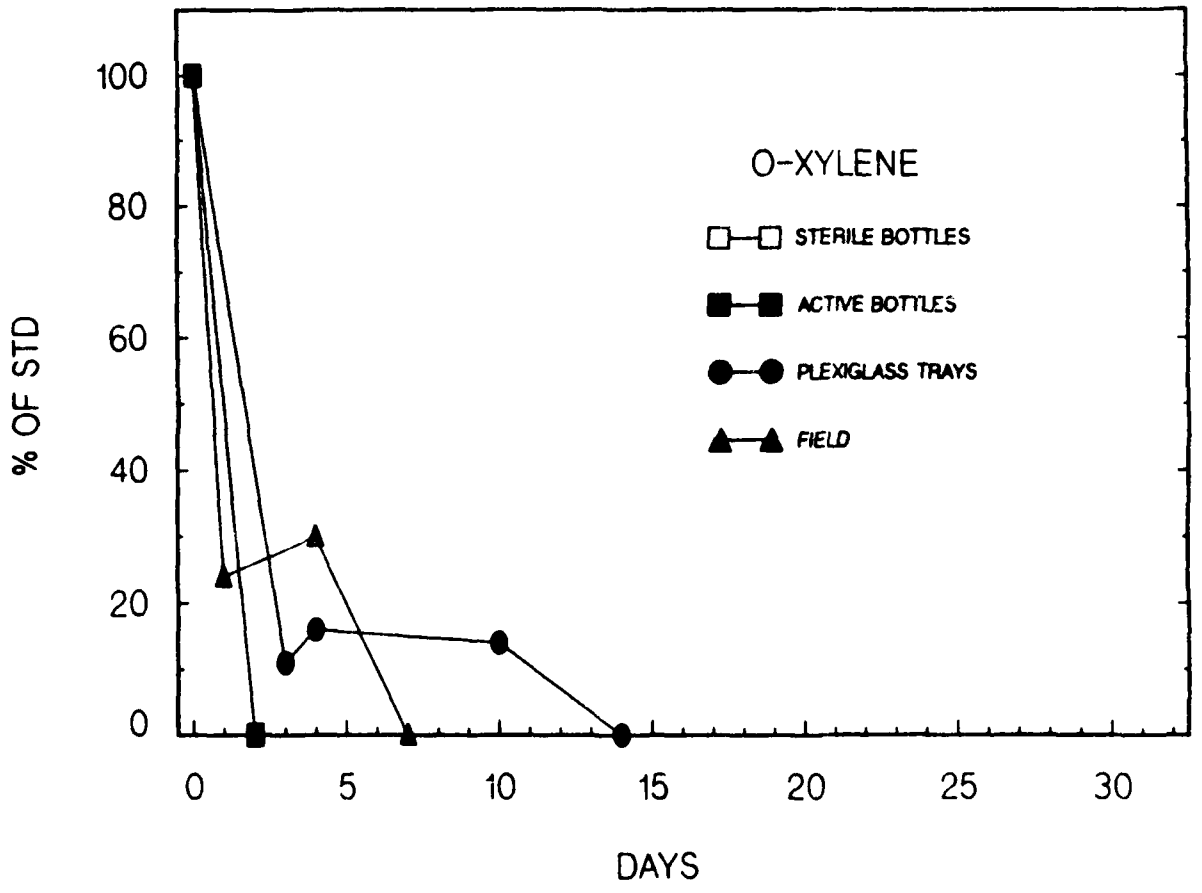



Figure 38. Change in Concentration Ratio (Expressed as Percent of Standard) of o-Xylene to Tetradecane in Samples Taken from the Deep Water Systems.

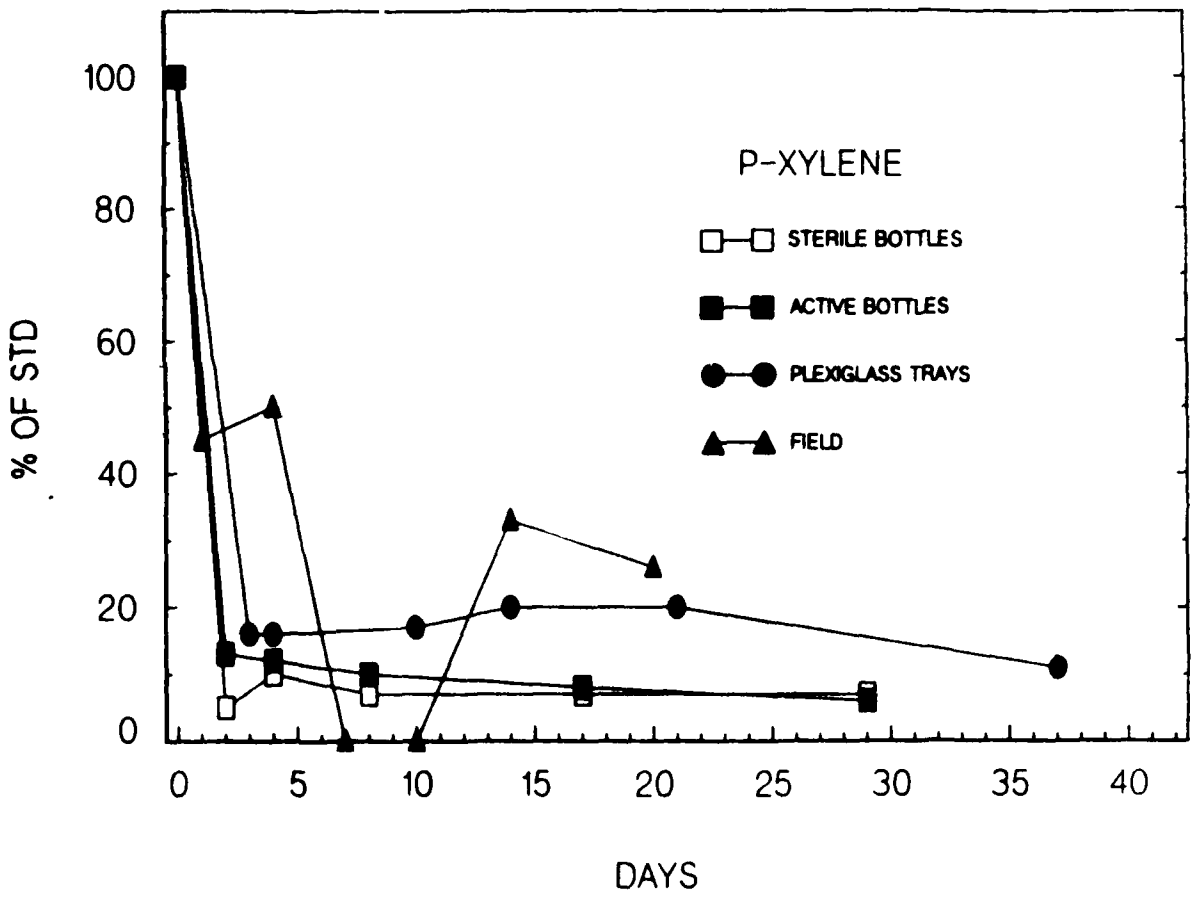



Figure 39. Change in Concentration Ratio (Expressed as Percent of Standard) of p-Xylene to Tetradecane in Samples Taken from the Deep Water Systems.

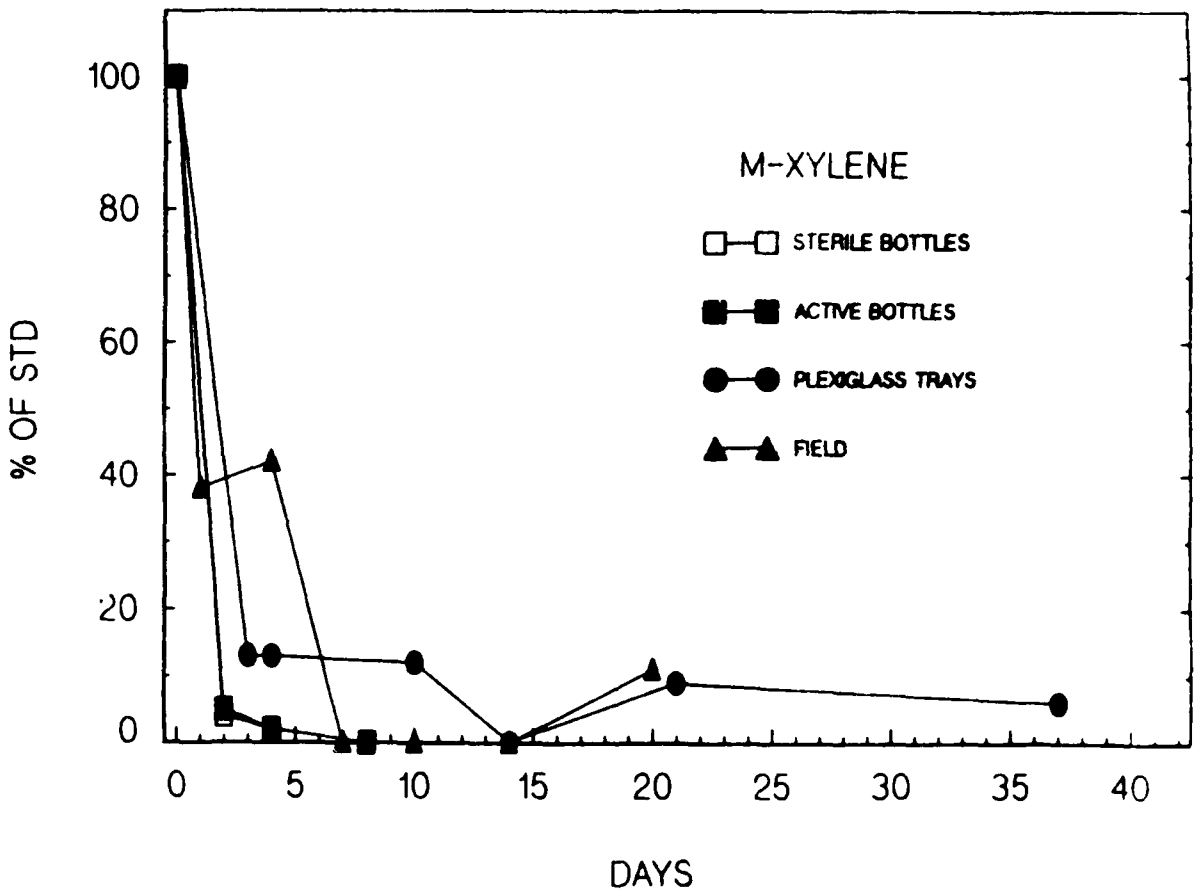



Figure 40. Change in Concentration Ratio (Expressed as Percent of Standard) of m-Xylene to Tetradecane in Samples Taken from the Deep Water Systems.

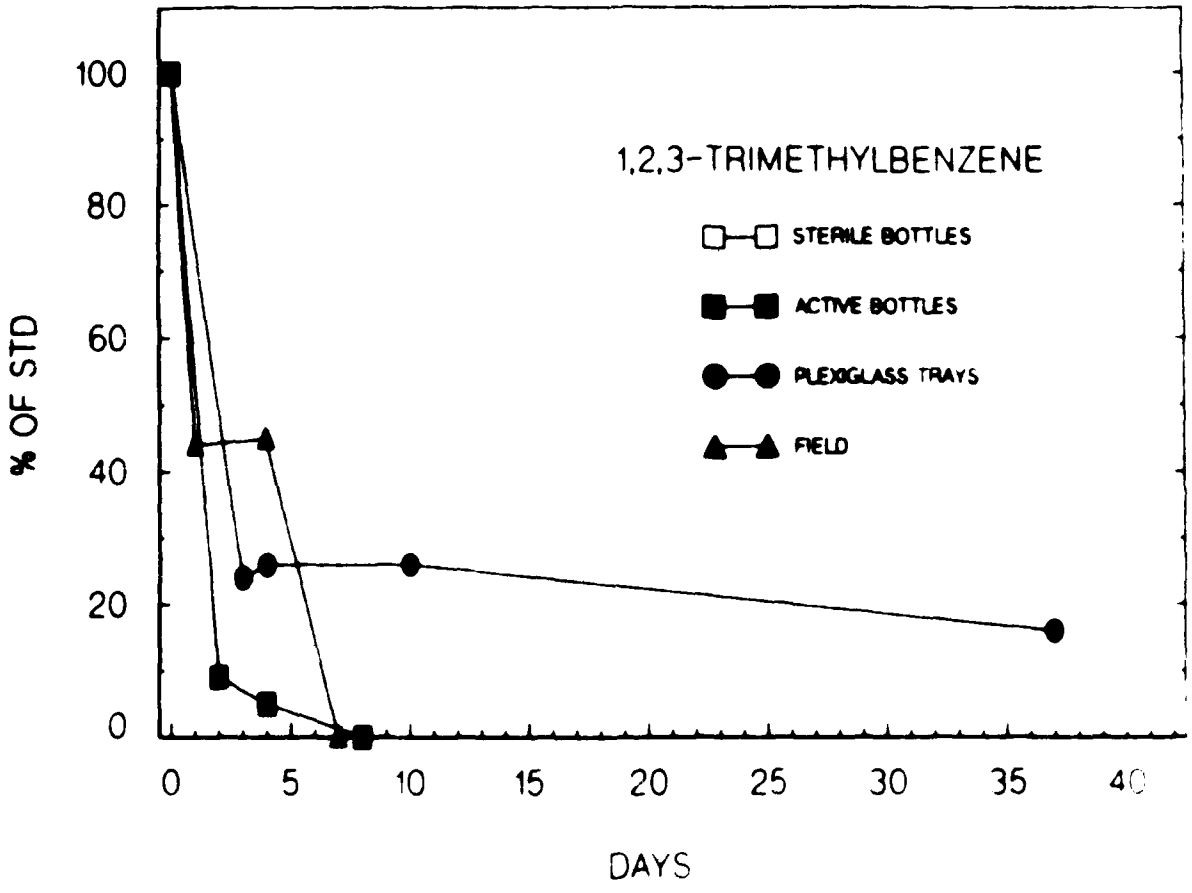



Figure 4i. Change in Concentration Ratio (Expressed as Percent of Standard) of 1,2,3-Trimethylbenzene to Tetradecane in Samples Taken from the Deep Water Systems.

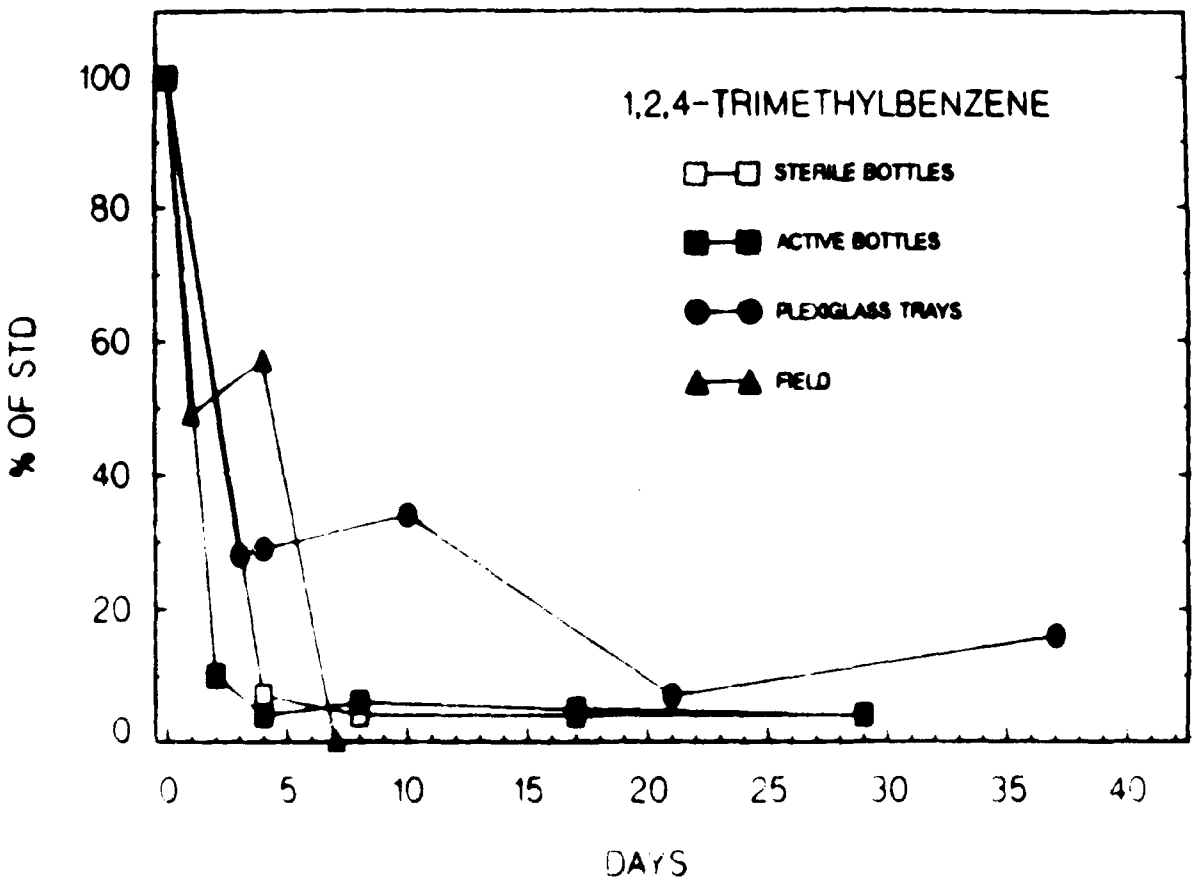



Figure 47. [illegible] expressed as percent of standard] [illegible] 1,2,4-trimethylbenzene [illegible] samples taken from the deep water system.

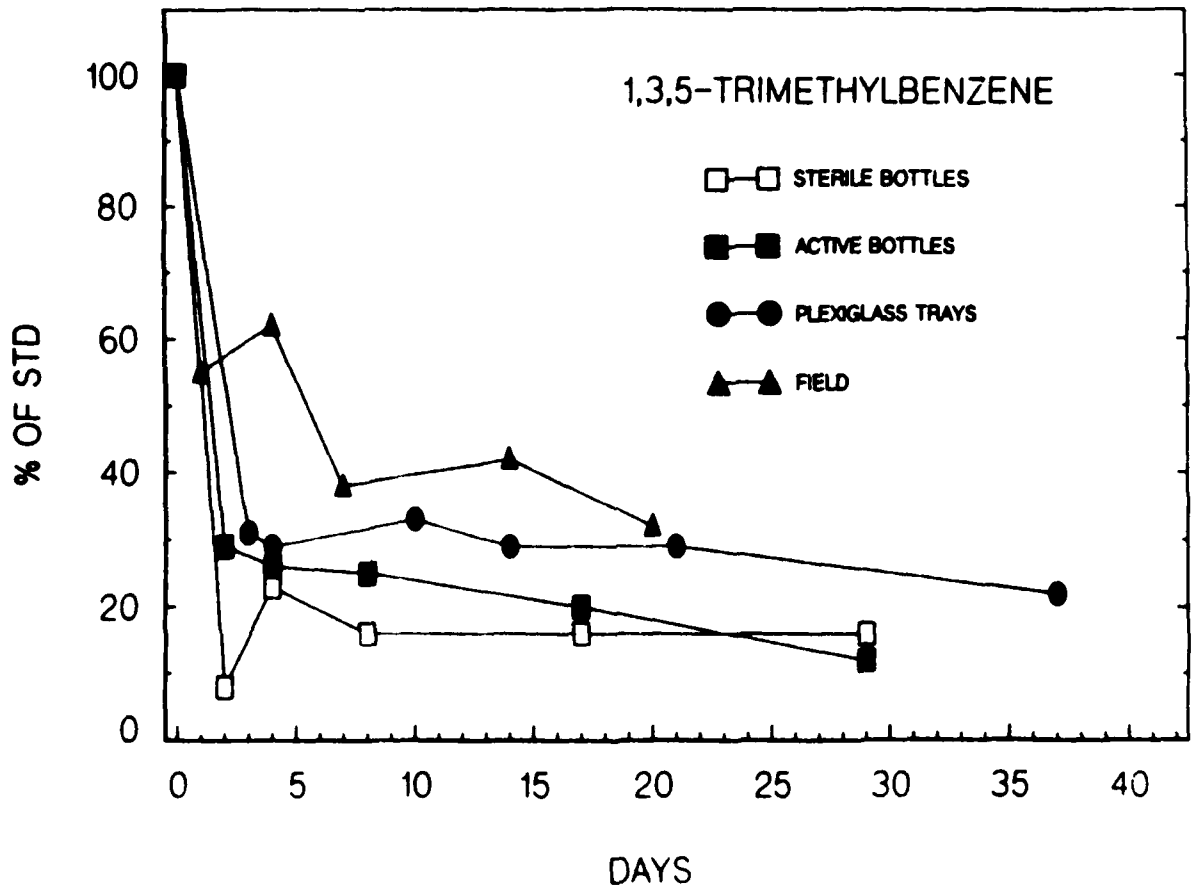



Figure 43. Change in Concentration Ratio (Expressed as Percent of Standard) of 1,3,5-Trimethylbenzene to Tetradecane in Samples Taken from the Deep Water Systems.

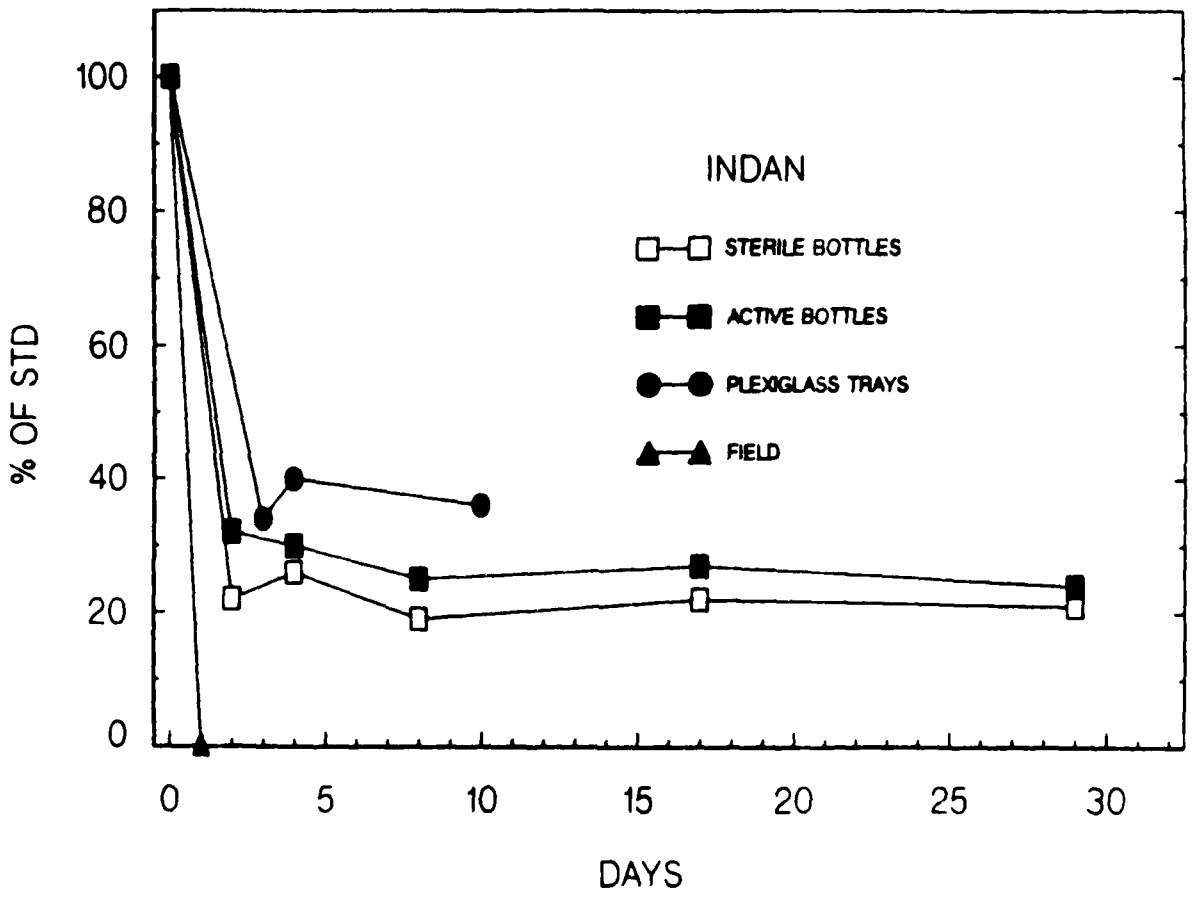



Figure 44. Change in Concentration Ratio (Expressed as Percent of Standard) of Indan to Tetradecane in Samples Taken from the Deep Water Systems.

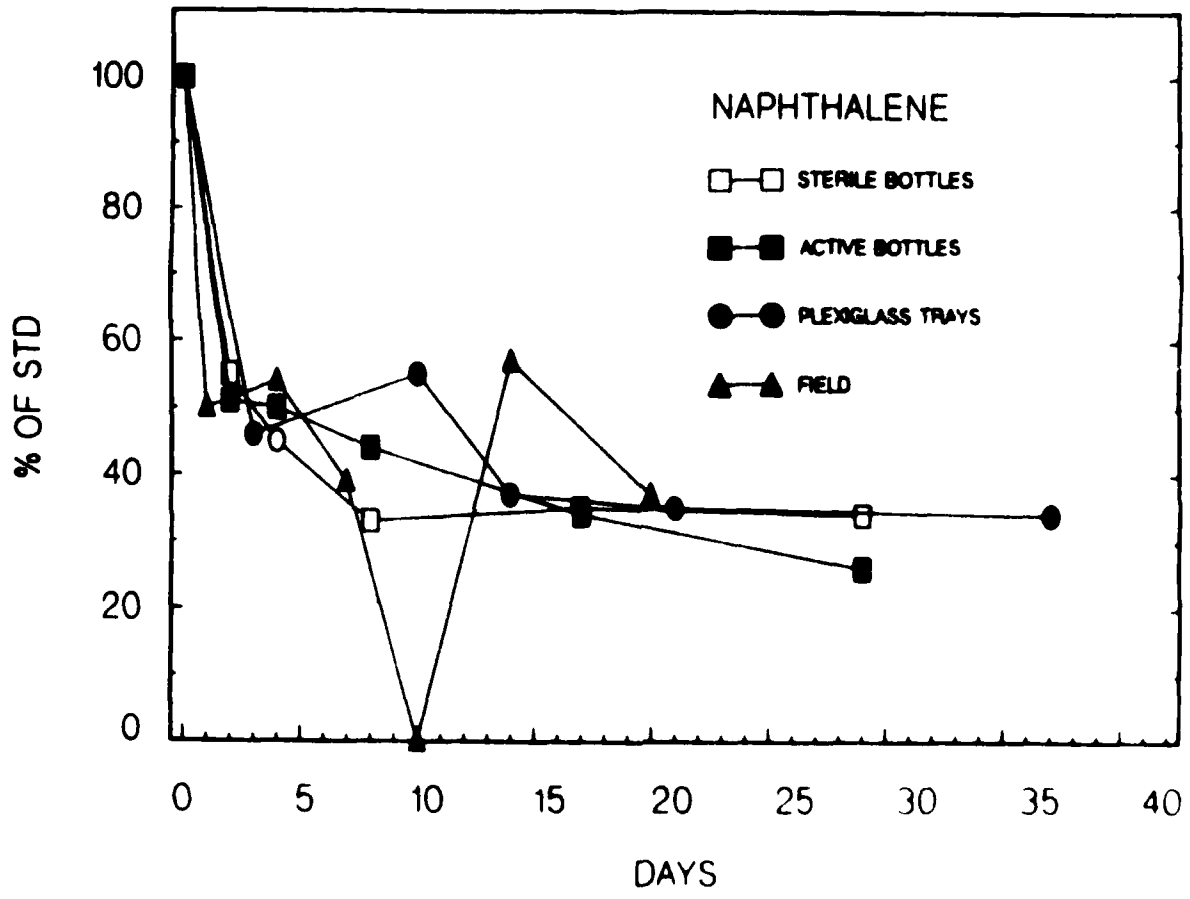
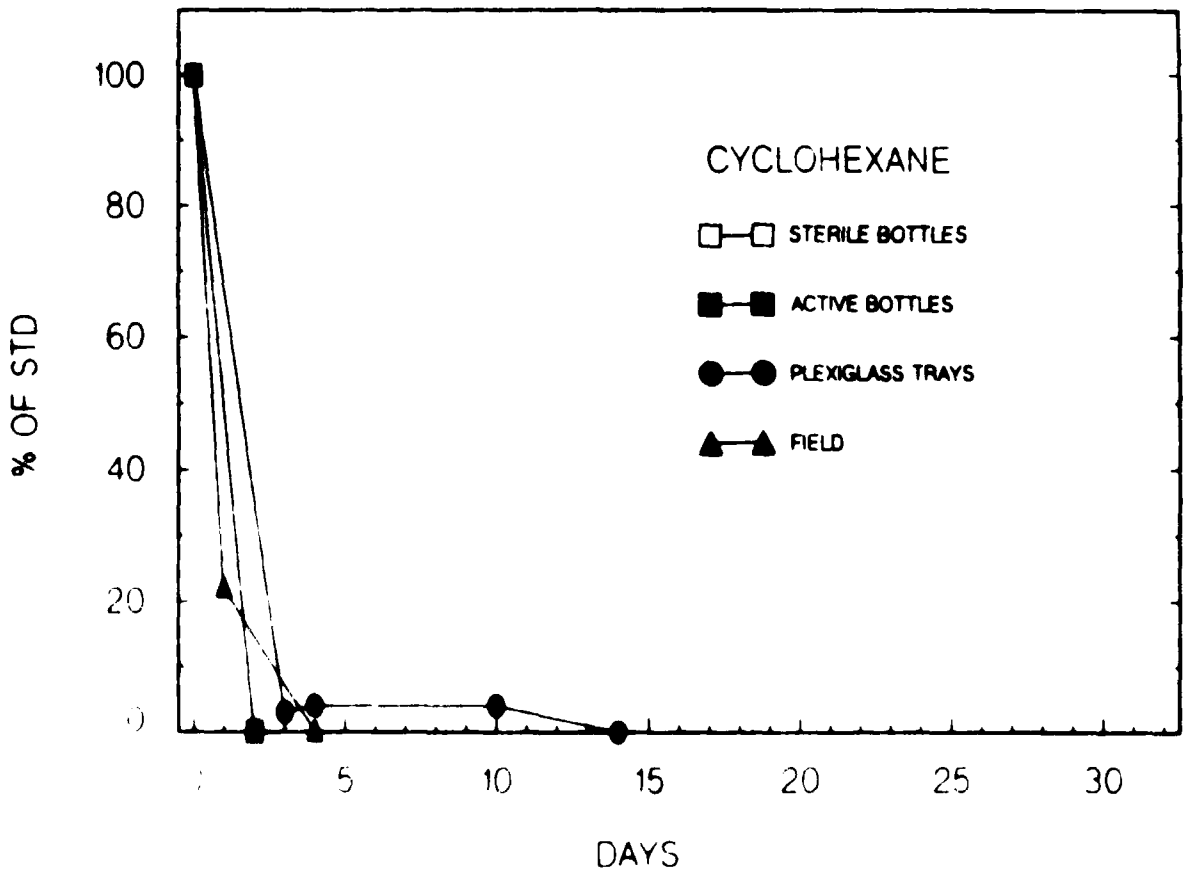



Figure 45. Change in Concentration Ratio (Expressed as Percent of Standard) of Naphthalene to Tetradecane in Samples Taken from the Deep Water Systems.




[illegible] Concentration Data (Expressed as Percent of Standard) for Cyclohexane and Tetradecane in Samples Taken from the Deep Water [illegible]

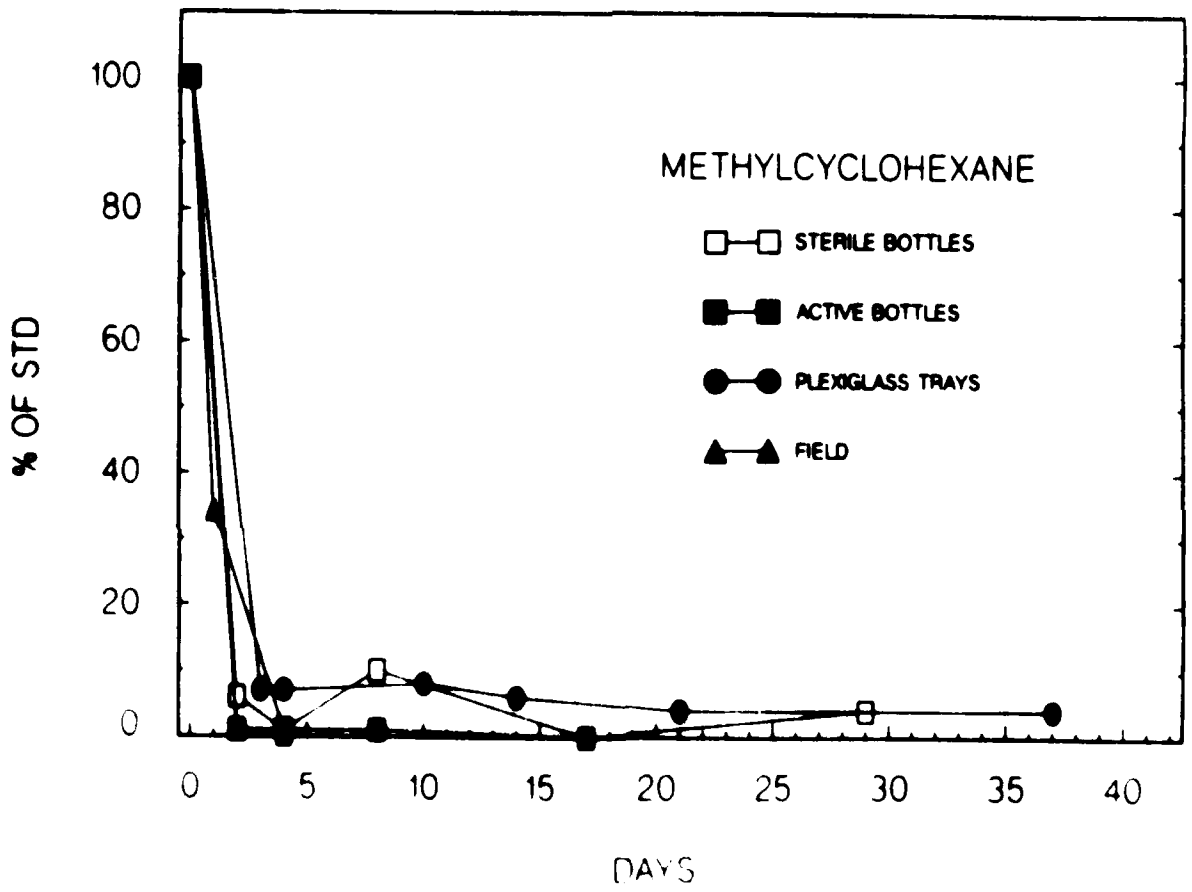



Figure 47. Change in Concentration Ratio (Expressed as Percent of Standard) of methylcyclohexane to Tetradecane in Samples Taken from the Deep Water Systems.

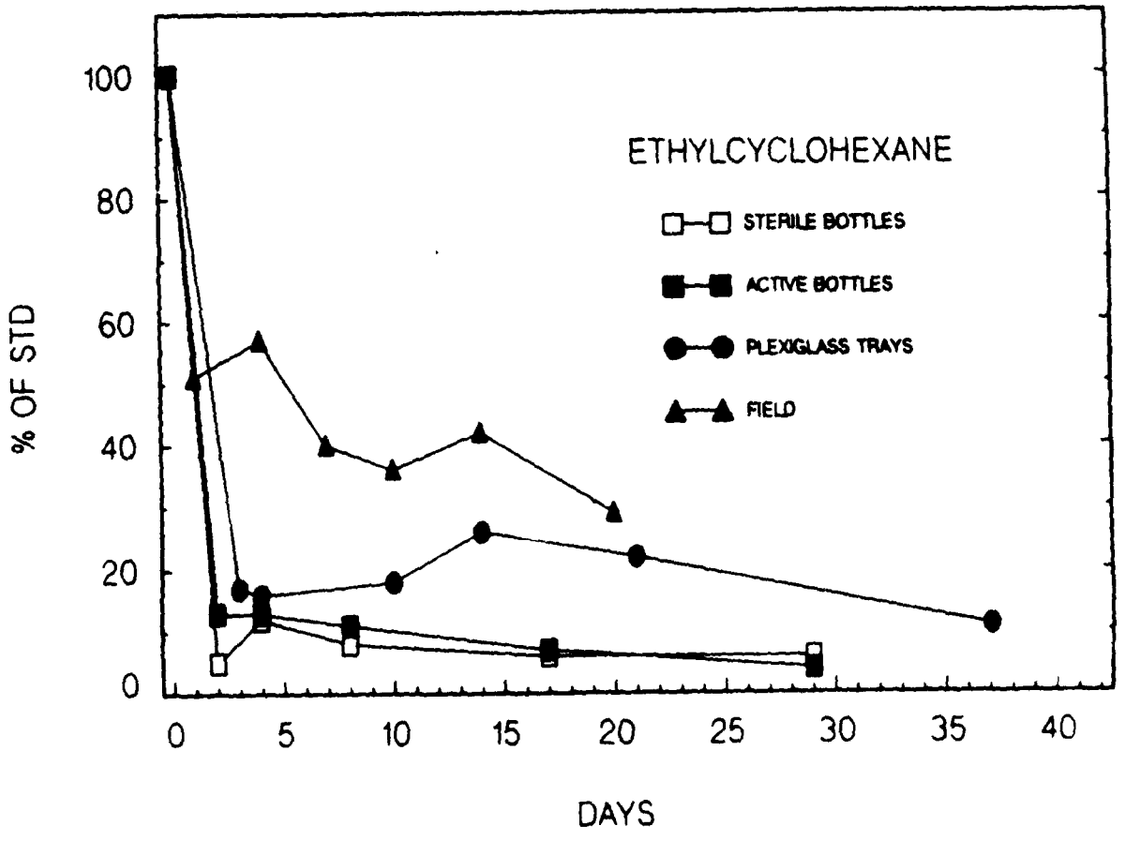



Figure 48. Change in Concentration Ratio (Expressed as Percent of Standard) of Ethylcyclohexane to Tetradecane in Samples Taken from the Deep Water Systems.

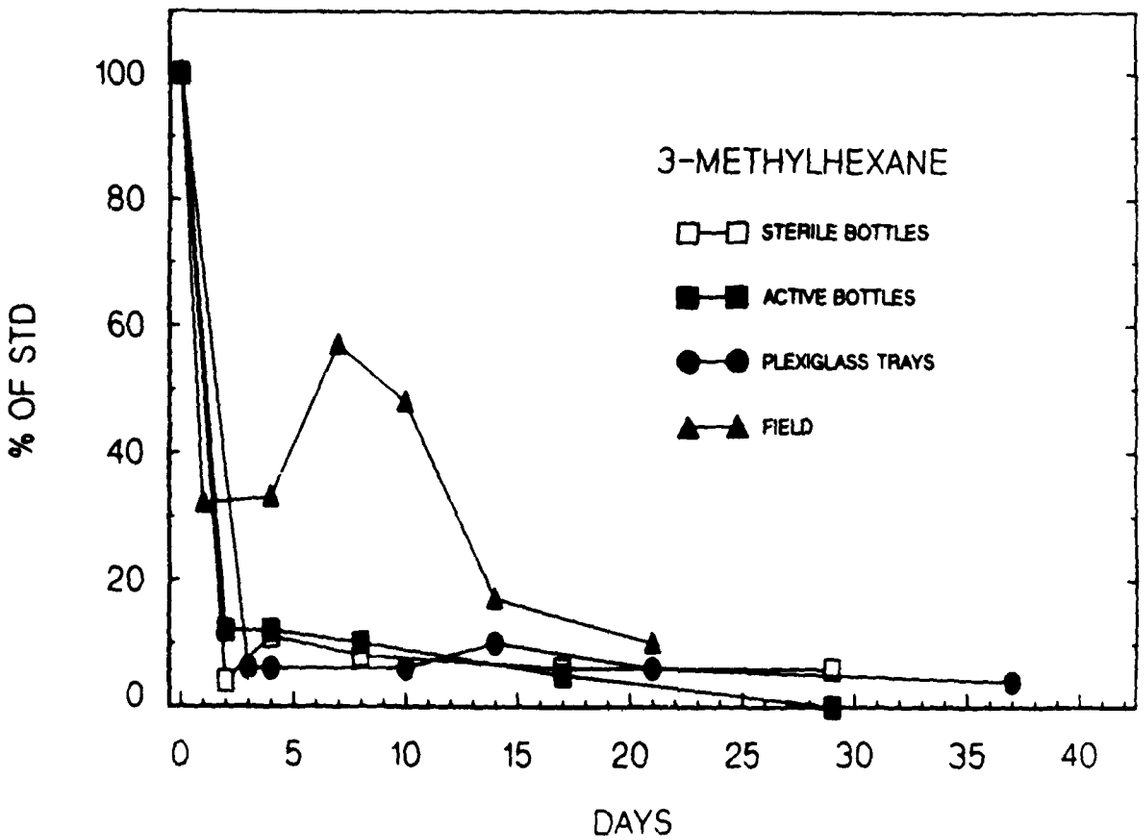



Figure 49. Change in Concentration Ratio (Expressed as Percent of Standard) of 3-Methylhexane to Tetradecane in Samples Taken from the Deep Water Systems.

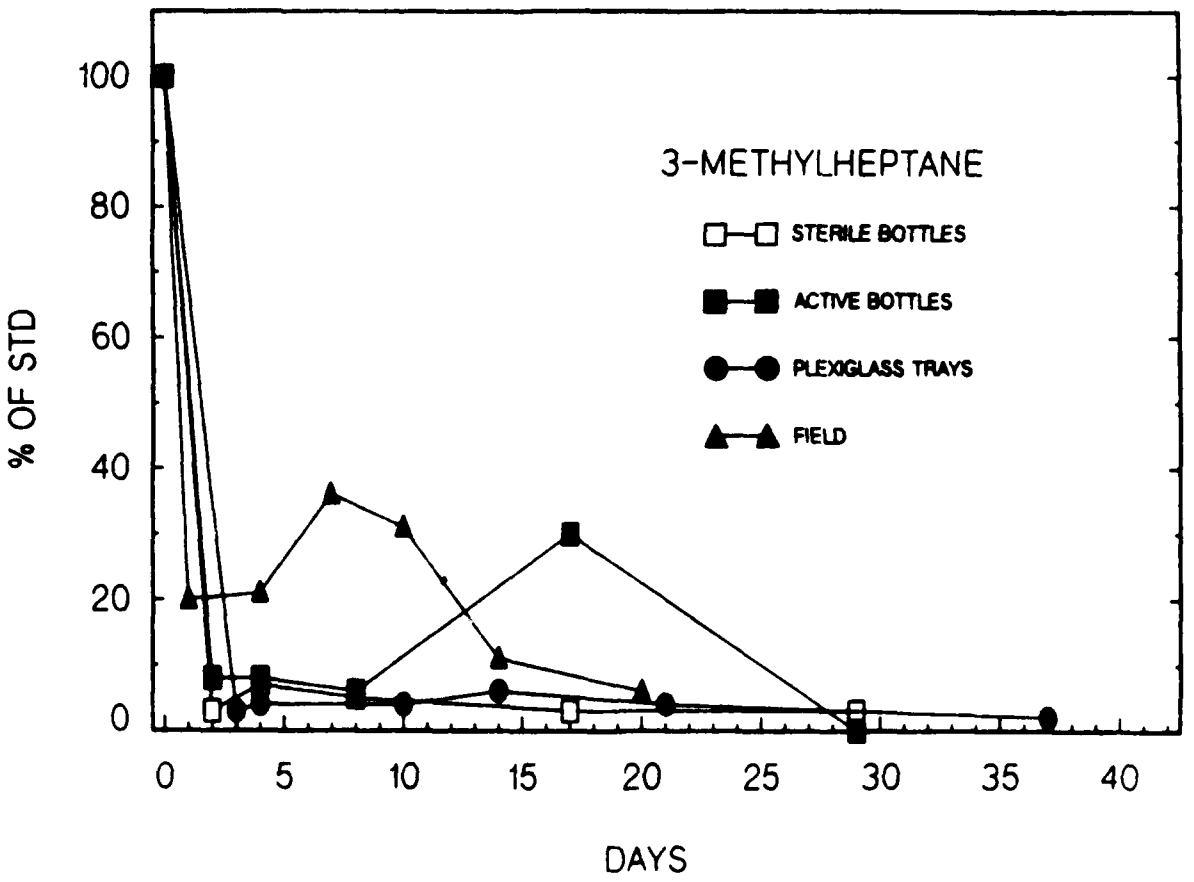



Figure 50. Change in Concentration Ratio (Expressed as Percent of Standard) of 3-Methylheptane to Tetradecane in Samples Taken from the Deep Water Systems.

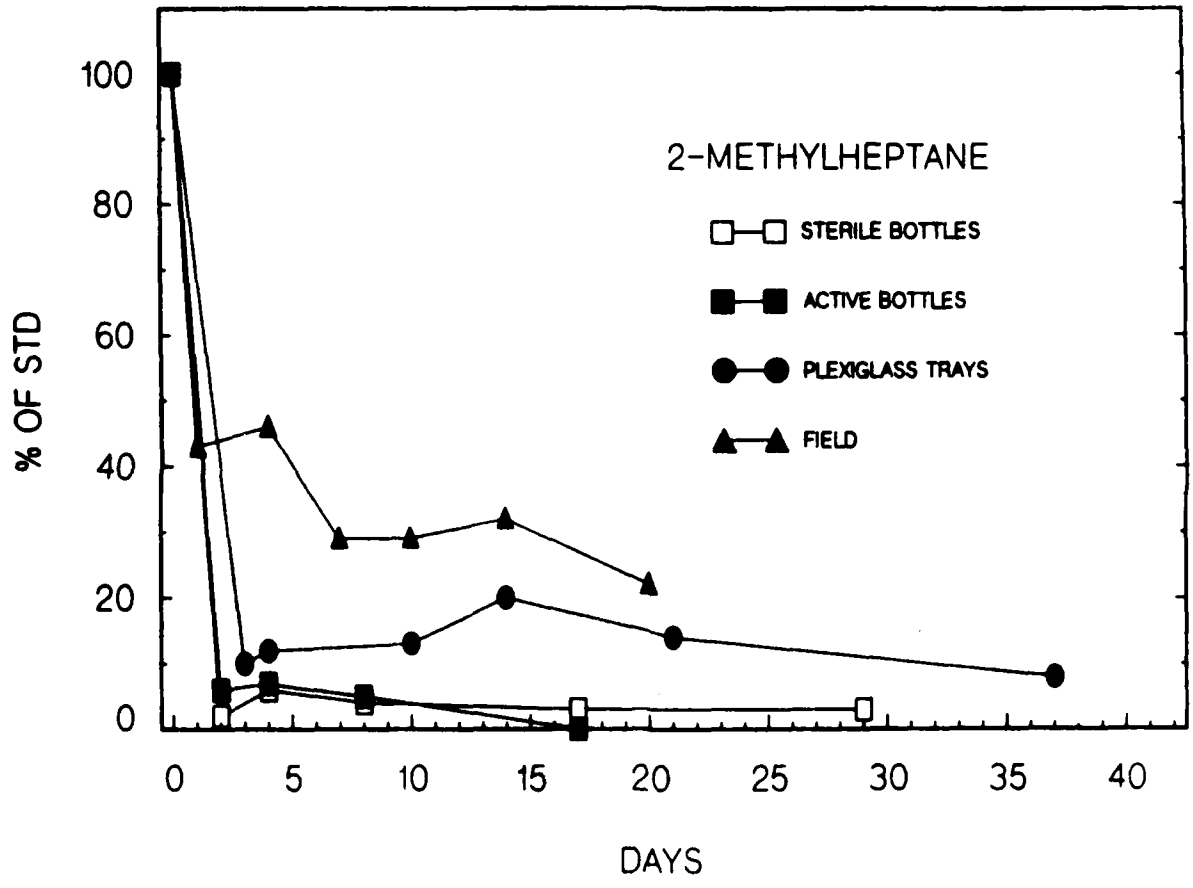



Figure 51. Change in Concentration Ratio (Expressed as Percent of Standard) of 2-Methylheptane to Tetradecane in Samples Taken from the Deep Water Systems.

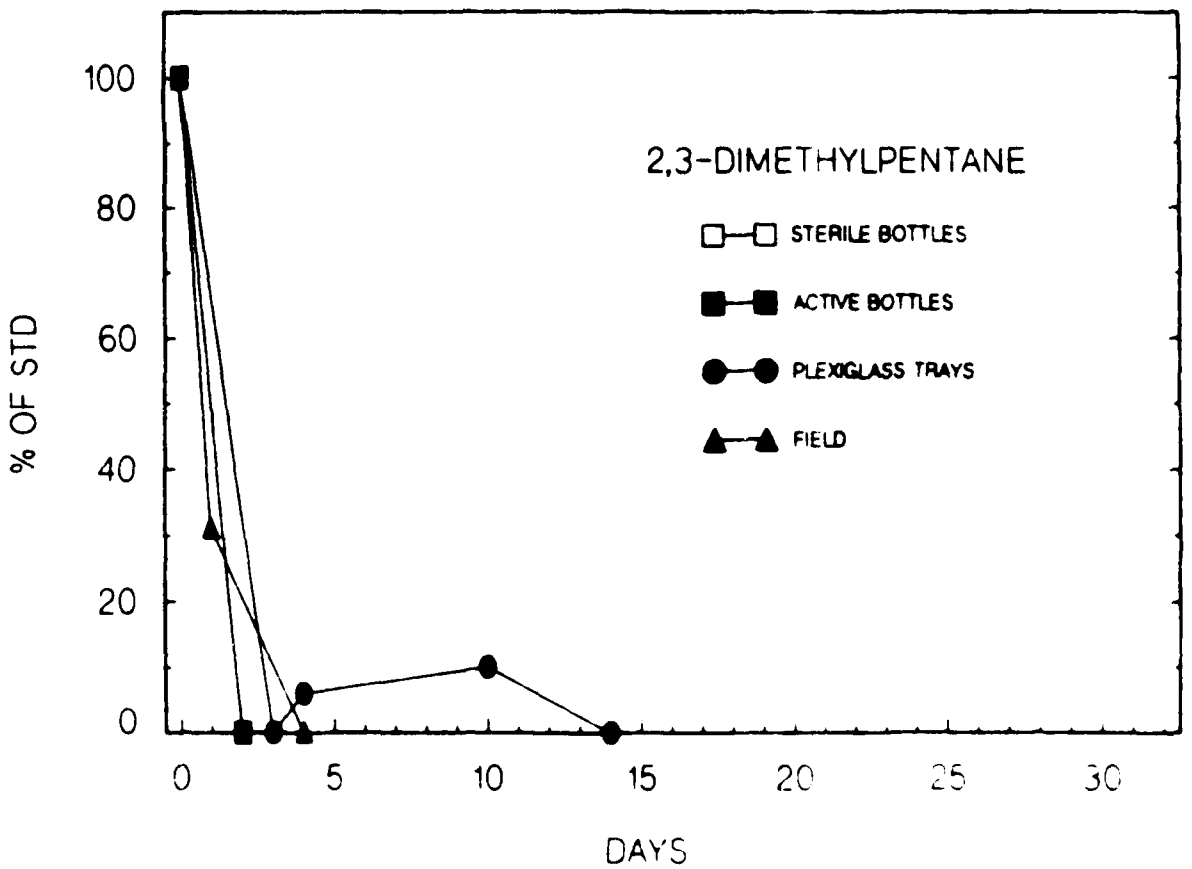



Figure 52. Change in Concentration Ratio (Expressed as Percent of Standard) of 2,3-Dimethylpentane to Tetradecane in Samples Taken from the Deep Water Systems.

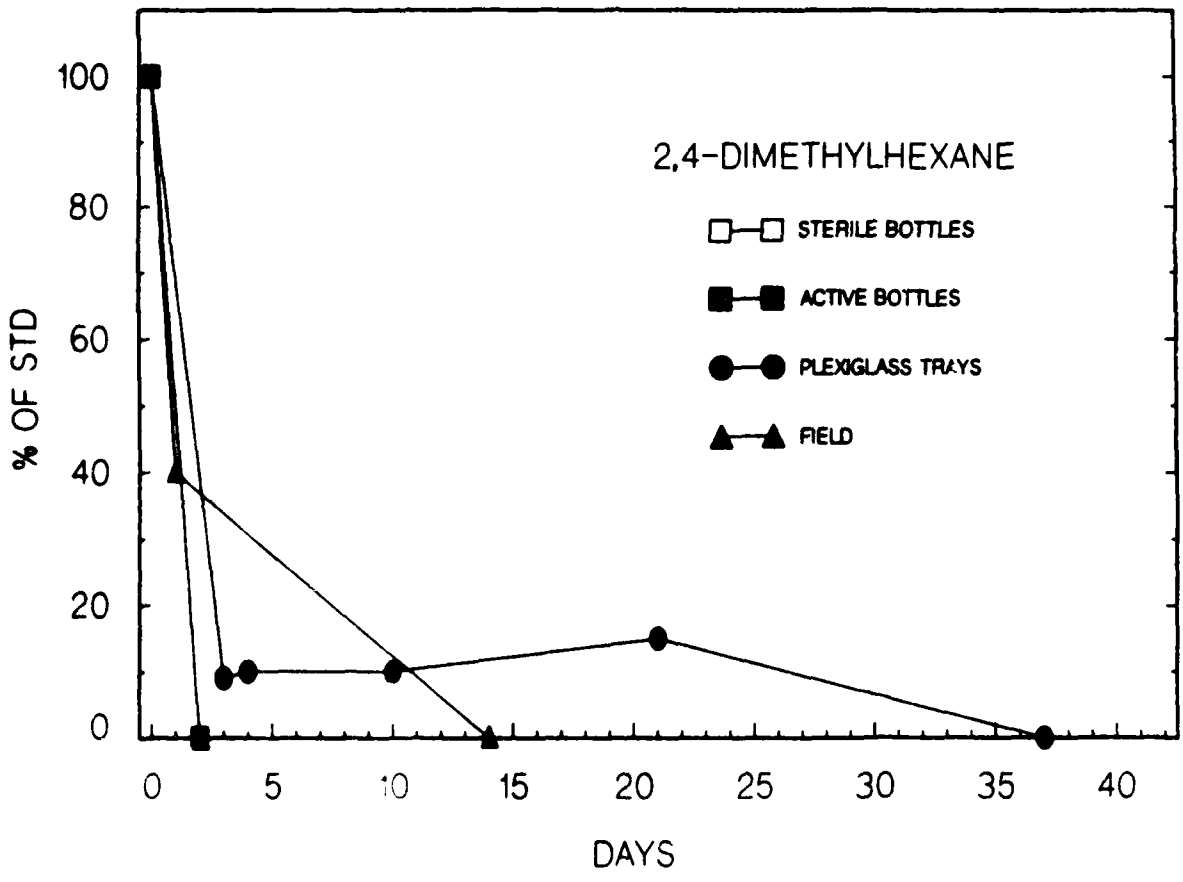
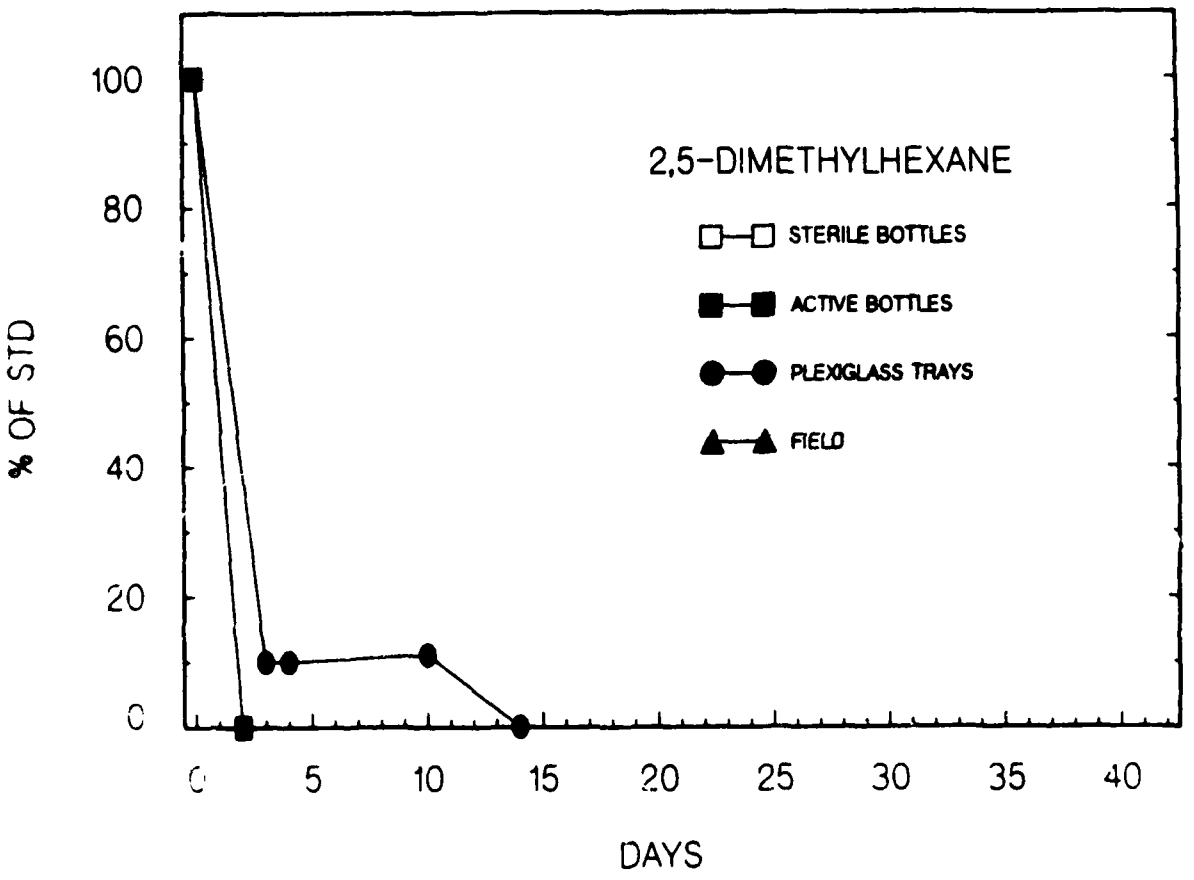



Figure 53. Change in Concentration Ratio (Expressed as Percent of Standard) of 2,4-Dimethylhexane to Tetradecane in Samples Taken from the Deep Water Systems.




...ge in Concentration Ratio (Expressed as Percent of Standard) ...,5-Dimethylhexane to Tetradecane in Samples Taken from the Deep ...ystems.

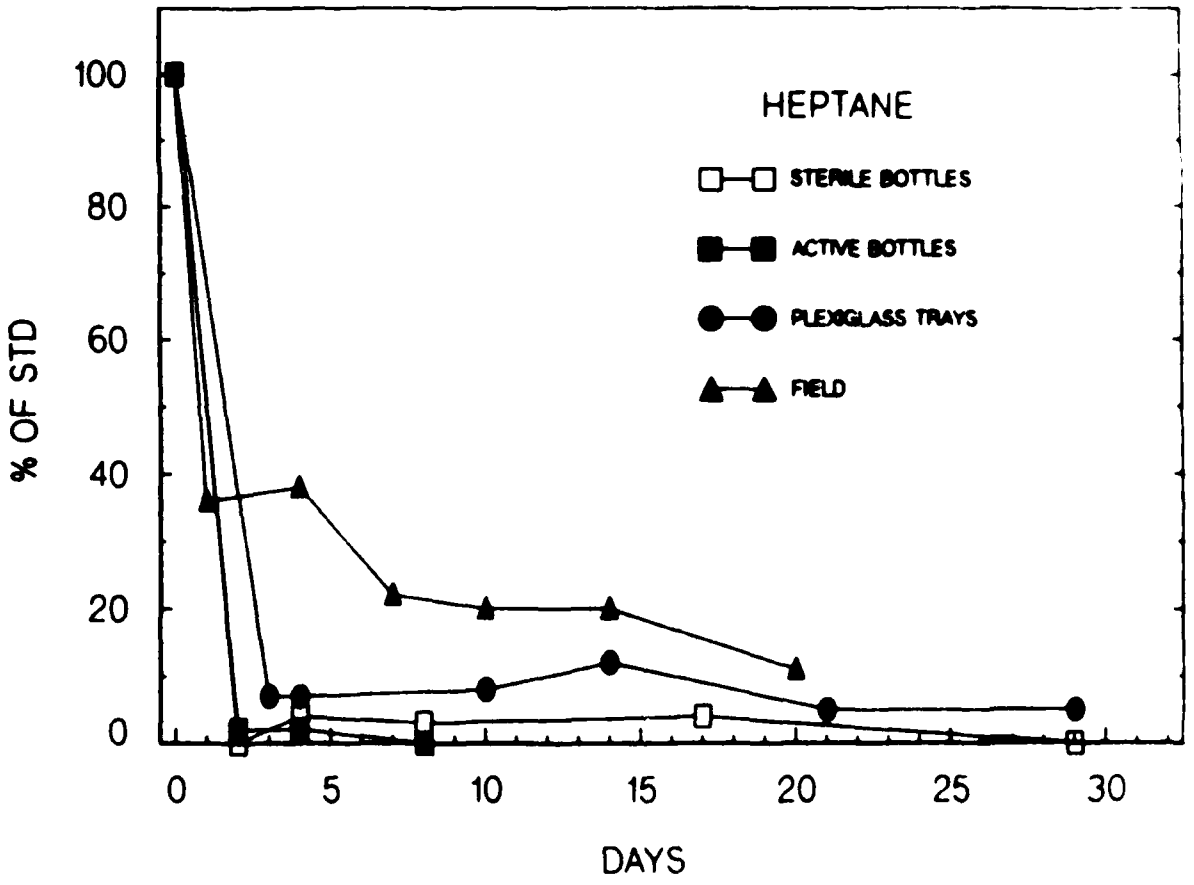



Figure 55. Change in Concentration Ratio (Expressed as Percent of Standard) of Heptane to Tetradecane in Samples Taken from the Deep Water Systems.

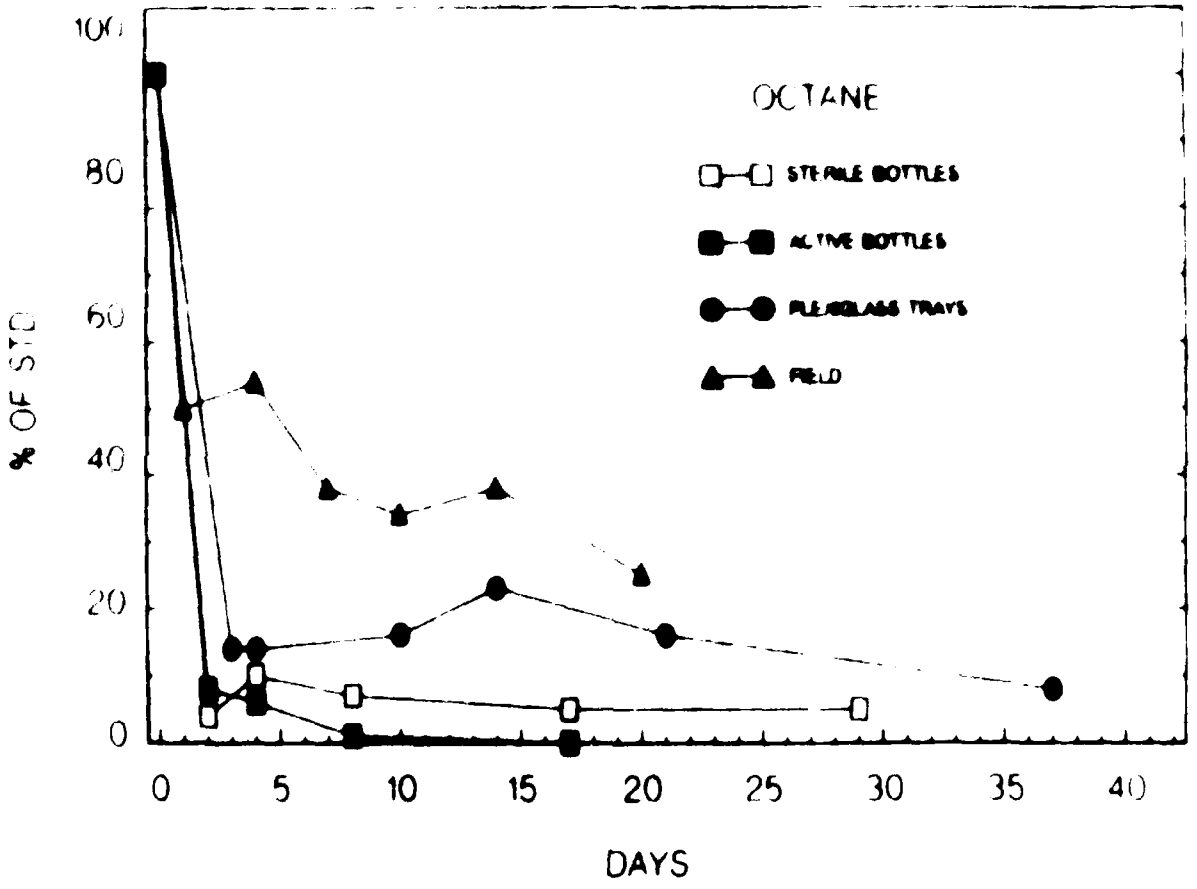



Figure 56. Change in Concentration Ratio (Expressed as Percent of Standard) of Octane to Tetradecane in Samples Taken from the Deep Water Systems.

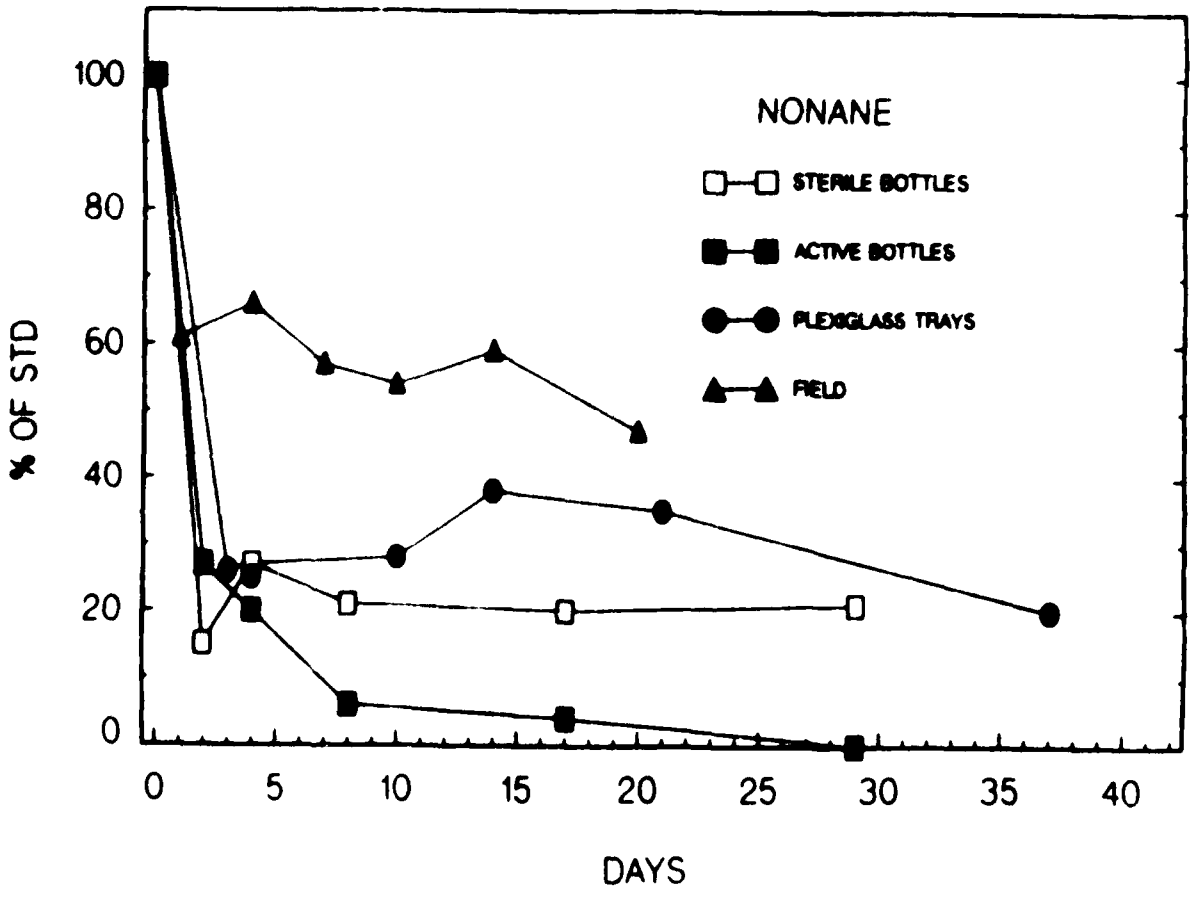



Figure 57. Change in Concentration Ratio (Expressed as Percent of Standard) of Nonane to Tetradecane in Samples Taken from the Deep Water Systems.

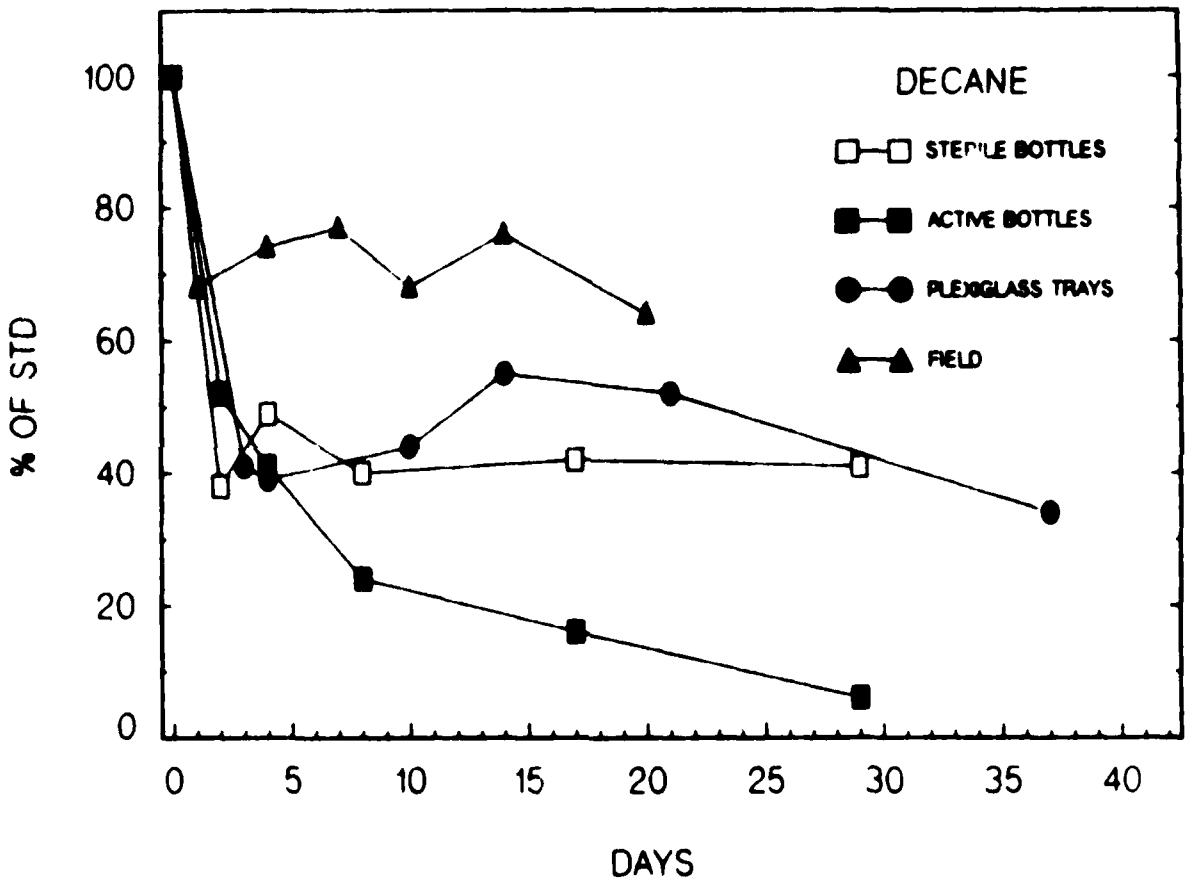



Figure 58. Change in Concentration Ratio (Expressed as Percent of Standard) of Decane to Tetradecane in Samples Taken from the Deep Water Systems.

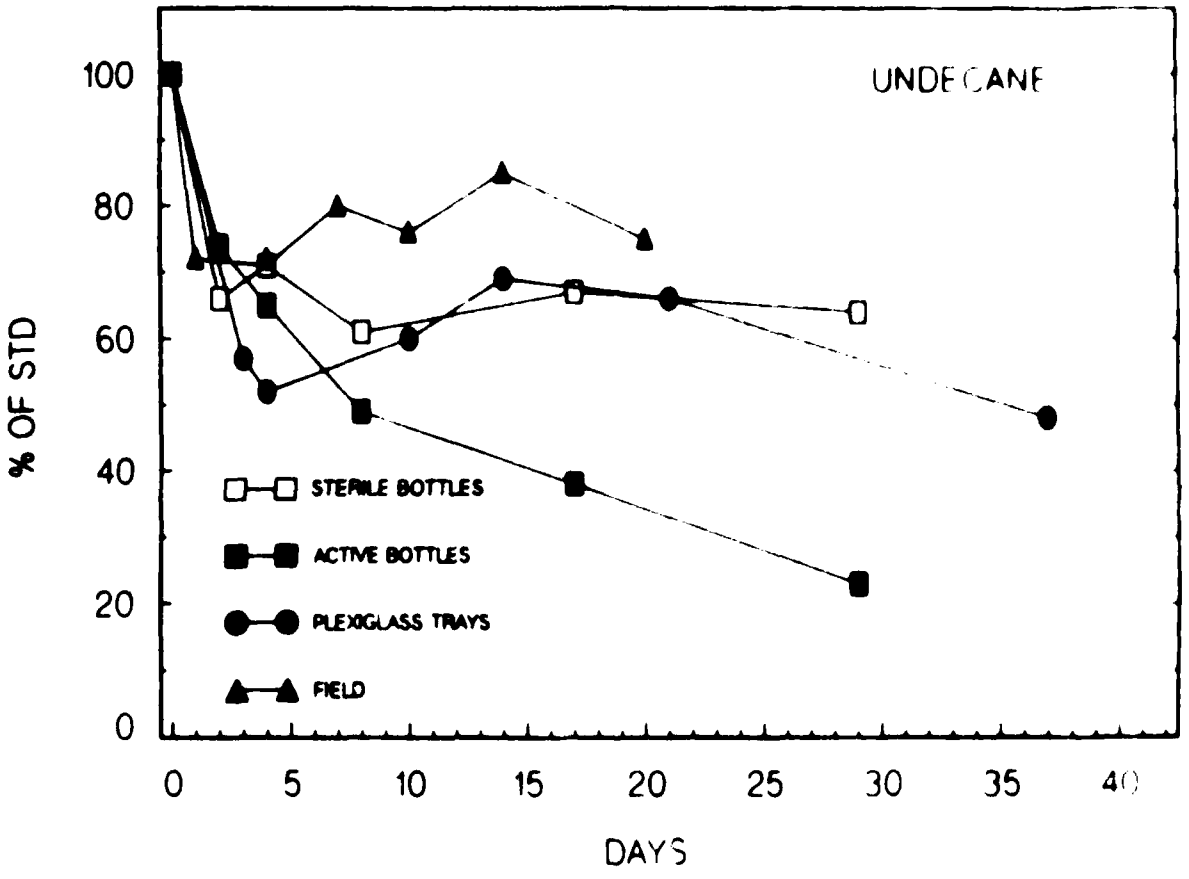



Figure 59. Change in Concentration Ratio (Expressed as Percent of Standard) of Undecane to Tetradecane in Samples Taken from the Deep Water Systems.

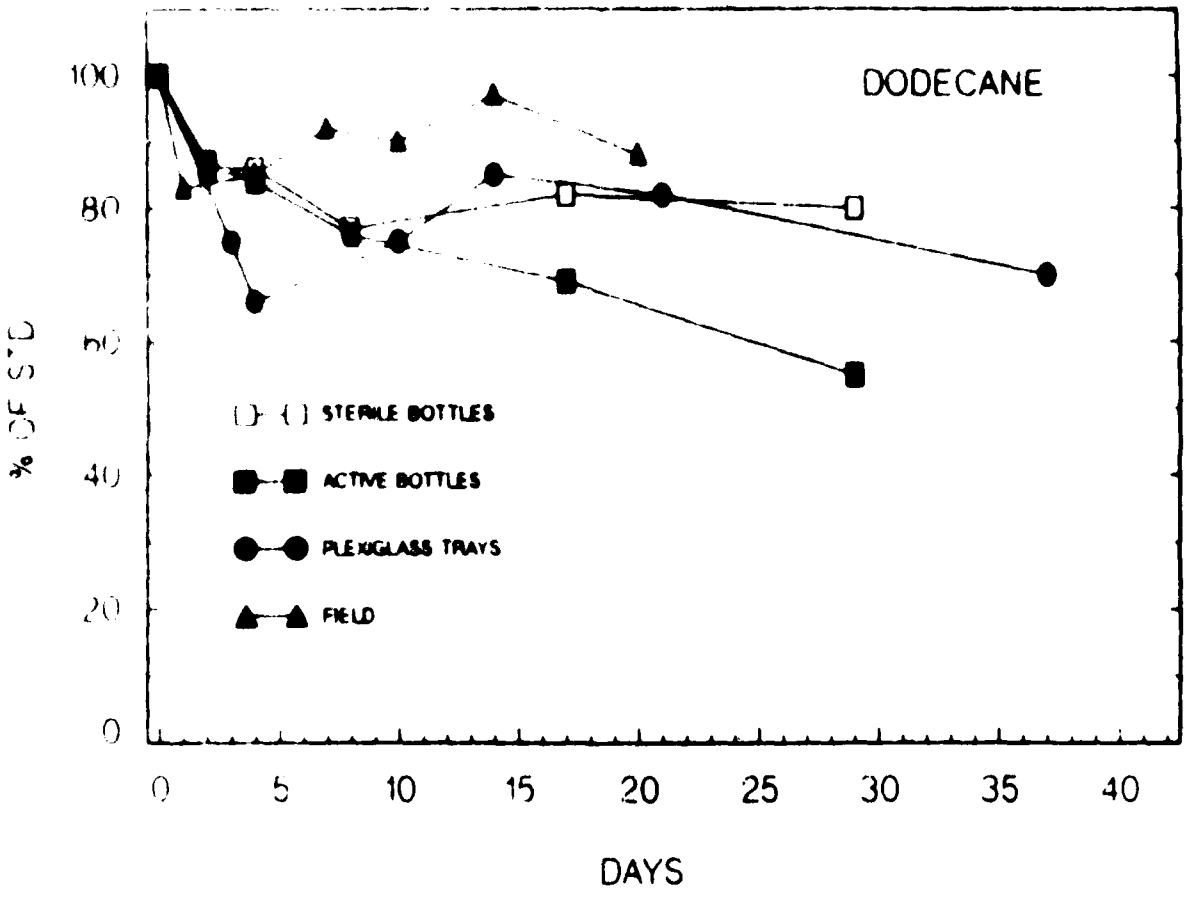



Figure 60 Change in Concentration Ratio (Expressed as Percent of Standard) of Dodecane to Tetradecane in Samples Taken from the Deep Water Systems.

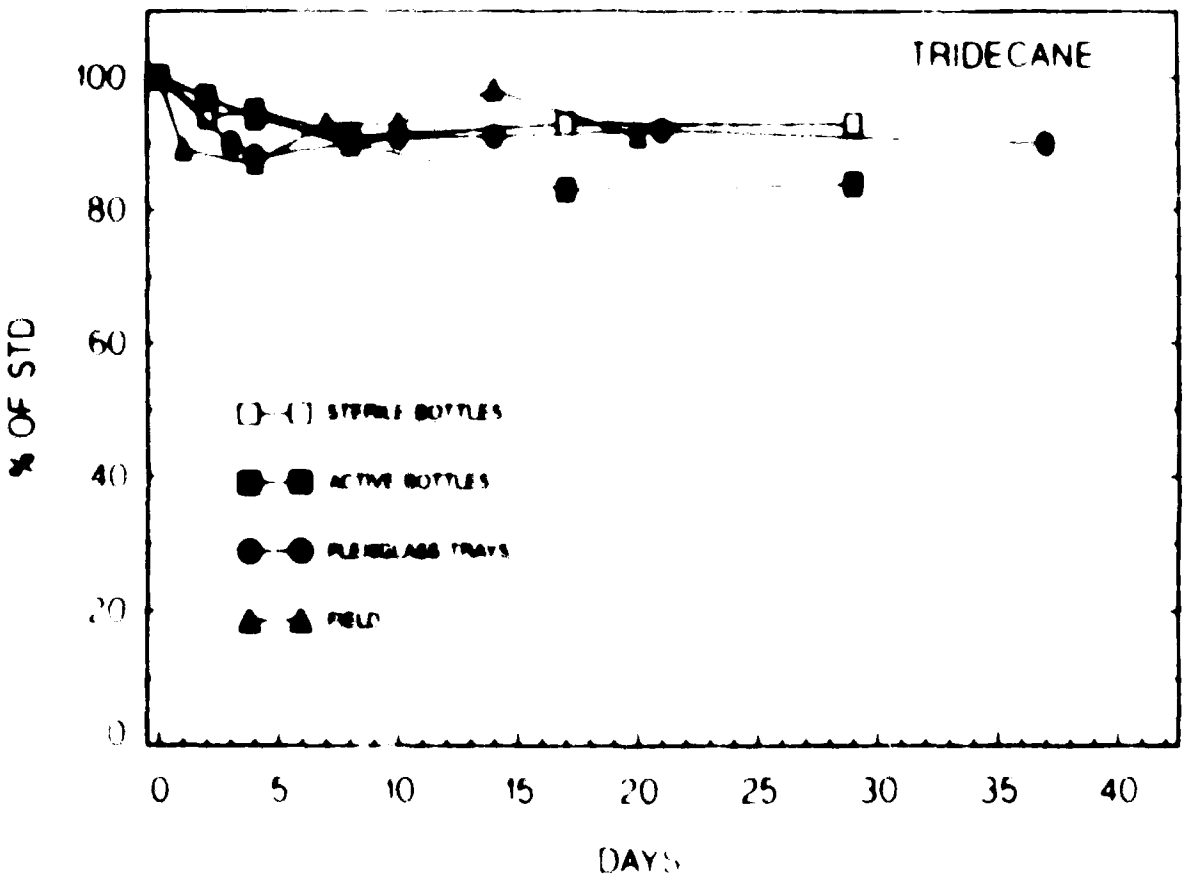



Figure 61. Change in Concentration Ratio (Expressed as Percent of Standard) of Tridecane to Tetradecane in Samples Taken from the Deep Water Systems.

A summary of hydrocarbon persistence in the bottle test is shown in Table 1. The majority of the hydrocarbons rapidly disappeared (ratios dropped below 10 percent of the standard within 5 days) from the bottles. Decreases in ratios for methyl and ethyl benzene were unusual in that one sampling with each hydrocarbon showed a ratio above 10 percent following the initial drop below 10 percent. Because each sampling period represents the analysis of a complete bottle, these discrepancies may have been the result of unintentionally incubating a bottle in a slightly different manner (e.g., less exposure to room air currents) from the others. The variability does not detract from the results which show that these hydrocarbons are not persistent.

Naphthalene, indan, 1,3,5-trimethylbenzene (TMB) and the normal alkanes (nonane-through-tridecane) were the most persistent hydrocarbons in the bottles tests, with ethyl-cyclohexane, 3-methylhexane, octane and nonane being somewhat intermediate, depending on the experimental conditions (i.e.,deep site simulation, nonsterile conditions).

Biodegradation was apparent for all of the normal alkanes except possibly possibly tridecane. For several of the hydrocarbons (1,3,5-TMB, indan, naphthalene, 2-and 3-methylheptanes, 3-methylhexane and tridecane) large differences in ratios between sterile and active systems occurred toward the end of the incubation period, suggesting the possible onset of biodegradation.

Although ratio values are quite low, there was also some indication that losses of several hydrocarbons (heptane, octane, nonane, p-xylene, 1,3,5-TMB, 1,2,3-TMB, 2-and 3-methylheptane, 3-methylhexane, and ethycyclohexane) were lower in the deep water test bottles than in the shallow water test bottles.

b. Field samples

A summary of the persistence of hydrocarbons in the field samples is shown in Table 2. In general, hydrocarbon disappearance was quite apparent despite some variability from one sampling period to another. Although attempts had been made to standardize our sampling procedures as much as possible, there was no way to guarantee that uncontaminated sediments would not dilute out the contaminated sediments during any particular sampling and thus, give very low hydrocarbon concentrations. This sampling problem was probably responsible for the frequently observed increase in hydrocarbon concentration over two successive sampling periods. Because this variability could not be controlled, we generally looked for overall qualitative trends and asked whether a hydrocarbon was either present or absent. The detection of any hydrocarbon in a sample seemed important since our extractions of uncontaminated sediment continually showed the absence of any peaks on the gas chromatograph which might cochromatograph with the hydrocarbon in question and give a false positive.

Some of the aromatic hydrocarbons, the dimethyl alkanes and the cyclohexanes did not persist beyond 5-7 days. Of the toxic aromatic hydrocarbons, the the trimethyl benzenes, some of the dimethyl benzenes (xylenes) and naphthalene all persisted for 10 days or more and in several cases were still detectable at the end of the experimental period.

The normal alkanes were the most persistent hydrocarbons, with undecane, dodecane, and tridecane showing essentially no losses over the incubation period.

TABLE 1. PERSISTENCE (RATIO VALUE GREATER THAN 10 PERCENT OF THE VALUE IN STANDARD) OF SPECIFIC HYDROCARBONS IN JP-4-CONTAMINATED SEDIMENTS USED IN BOTTLE TESTS.

| | Day When Ratio Value Decreased Below 10% of Standard | | | | |
|---|---|---|---|---|---|
| **AROMATICS** | 5 | 10 | 15 | 20 | > 20 |
| methylbenzene[a] | x | | | | |
| ethylbenzene[a] | x | | | | |
| isopropylbenzene | x | | | | |
| o-xylene | x | | | | |
| p-xylene | x | | | | |
| m-xylene | x | | | | |
| 1,2,3-TMB[b] | x | | | | |
| 1,2,4-TMB | x | | | | |
| 1,3,5-TMB | | | | | x |
| indan | | | | | x |
| naphthalene | | | | | x |
| **CYCLIC ALKANES** | | | | | |
| cyclohexane | x | | | | |
| methylcyclohexane | x | | | | |
| ethylcyclohexane | x(S)[c] | x(D)[c] | | | |
| dimethylcyclohexanes | x | | | | |
| **BRANCHED ALKANES** | | | | | |
| 3 methylhexane | x(S) | x(D) | | | |
| 3 methylheptane | x | | | | |
| 2 methylheptane | x | | | | |
| dimethylheptanes | x | | | | |

TABLE 1. PERSISTENCE (RATIO VALUE GREATER THAN 10 PERCENT OF THE VALUE IN STANDARD) OF SPECIFIC HYDROCARBONS IN JP-4-CONTAMINATED SEDIMENTS USED IN BOTTLE TESTS (CONCLUDED).

| | Day When Ratio Value Decreased Below 10% of Standard | | | | |
|---|---|---|---|---|---|
| NORMAL ALKANES | 5 | 10 | 15 | 20 | > 20 |
| heptane | x | | | | |
| octane | x(S) | x(D) | | | |
| nonane | | a(S) | | | x(D) |
| decane | | | | | x |
| undecane | | | | | x |
| dodecane | | | | | x |
| tridecane | | | | | x |

---

[a] data variable

[b] TMB, trimethylbenzene

[c] S, bottle simulating shallow water condition; D, bottle simulating deep water condition

The monosubstituted alkanes were also quite persistent, as was ethylcyclohexane.

Many of the hydrocarbons (p-xylene, 1,3,5-TMB, naphthalene, the monosubstituted alkanes, heptane, octane, nonane and ethylcyclohexane) showed considerably greater persistence at the deep water site than at the shallow water site. Site differences in persistence for decane, undecane, dodecane, and tridecane were not apparent due to their slow rates of disappearance. Most of the other hydrocarbons were likewise lost too rapidly to detect differences according to site.

c. Plexiglass Trays

Data from the tray experiments were erratic and difficult to associate with a particular trend. In general, however, most of the jet fuel hydrocarbons associated with the sediment in the trays were detectable at low concentrations (bordering on the limits of detection) up until the end of the incubation period. The erratic results could have been caused by unavoidable inconsistencies in sampling; that is, the sampling procedure probably caused the sediment to redistribute unevenly in the trays. In fact, actual concentrations of both the volatile and nonvolatile hydrocarbons varied considerably from one sampling to another suggesting the problems in sampling rather than problems in sample handling which could cause loss of just the volatile hydrocarbons.

However, despite these inconsistencies, it would appear that most of the hydrocarbons persisted at low concentrations in the trays for considerable periods; i.e., their mere presence at any sampling period suggested they were slow to degrade or volatilize. Hydrocarbon concentrations, in many cases, dropped below 10 percent within 5 days. Only ethylcyclohexane, all normal alkanes (expect heptane), 2-methylheptane heptane, 1,3,5-TMB and naphthalene were found consistently above 10 percent throughout most of the incubation period. Several of the aromatic hydrocarbons (ethylbenzene, isopropylbenzene, o-xylene, p-xylene, m-xylene 1,2,3-TMB, 1,2,4-TMB, indan) were generally found above 10 percent for about 15 to 20 days, particularly in the trays kept in the deep water site. In addition, cyclohexane, dimethylcyclohexane, the dimethylheptanes, isopropylbenzene, o-xylene, and 1,2,3-TMB, all hydrocarbons which showed very rapid loss in the field sites and in the bottle tests, seemed to persist longer in the trays.

d. Toxicity

Toxicity tests with mysid shrimp, blue crabs, and minnows were conducted in the field during the dosing. Failure of control animals to survive, however, invalidated test results and no conclusions about toxicity of the JP-4 could be derived.

## D. DISCUSSION

### 1. Jet Fuel Hydrocarbon Fate

The results of our field study strongly suggest that if jet fuel becomes extensively mixed with natural sediment, many of the hydrocarbons contained therein will persist in the field, some for as long as 20 days or more. Similar

TABLE 2. PERSISTENCE (RATIO VALUE GREATER THAN 10 PERCENT OF THE VALUE IN STANDARD) OF SPECIFIC HYDROCARBONS IN JP-4-CONTAMINATED SEDIMENTS USED IN THE FIELD STUDY.

| | Day When Ratio Value Decreased Below 10% of Standard | | | | | |
|---|---|---|---|---|---|---|
| AROMATICS | <10 | 10 | 15 | 20 | >30 | No decrease |
| methylbenzene | x | | | | | |
| ethylbenzene | x | | | | | |
| isopropylbenzene | x | | | | | |
| o-xylene | x | | | | | |
| p-xylene | | x(S)a | | | x(D)a | |
| m-xylene | x(D) | x(S) | | | | |
| 1,2,3-TMB | | x(D) | x(S) | | | |
| 1,2,4-TMB | | x(D) | x(S) | | | |
| 1,3,5-TMB | | | | x(S) | x(D) | |
| indan | x | | | | | |
| naphthalene | | | | x(S) | x(D) | |
| CYCLIC ALKANES | | | | | | |
| cyclohexane | x | | | | | |
| methylcyclohexane | x(S) | x(D) | | | | |
| ethylcyclohexane | | | | x(S) | x(D) | |
| dimethylcyclohexanes | x | | | | | |
| BRANCHED ALKANES | | | | | | |
| 3-methylhexane | | | | x(S) | x(D) | |
| 3-methylheptane | | | | x(S) | x(D) | |
| 2-methylheptane | | | | x(S) | x(D) | |
| dimethylheptanes | x | | | | | |

TABLE 2. PERSISTENCE (RATIO VALUE GREATER THAN 10 PERCENT OF THE VALUE IN STANDARD) OF SPECIFIC HYDROCARBONS IN JP-4-CONTAMINATED SEDIMENTS USED IN THE FIELD STUDY (CONCLUDED).

| | Day When Ratio Value Decreases Below 10% of Standard | | | | | |
|---|---|---|---|---|---|---|
| | <10 | 10 | 15 | 20 | >20 | No decrease |
| **NORMAL ALKANES** | | | | | | |
| heptane | | x(S) | | | x(D) | |
| octane | | | | x(S) | x(D) | |
| nonane | | | | | x | |
| decane | | | | | x | |
| undecane | | | | | | x |
| dodecane | | | | | | x |
| tridecane | | | | | | x |

[a] data variable

[b] TMB, trimethylbenzene

[c] S, shallow water site; D, deep water site

AD-A180 065 DEGRADATION OF JET AND MISSILE FUELS BY AQUATIC MICROBIAL COMMUNITIES(U) ENVIRONMENTAL RESEARCH LAB GULF BREEZE FL P H PRITCHARD ET AL. JUL 87 2/2

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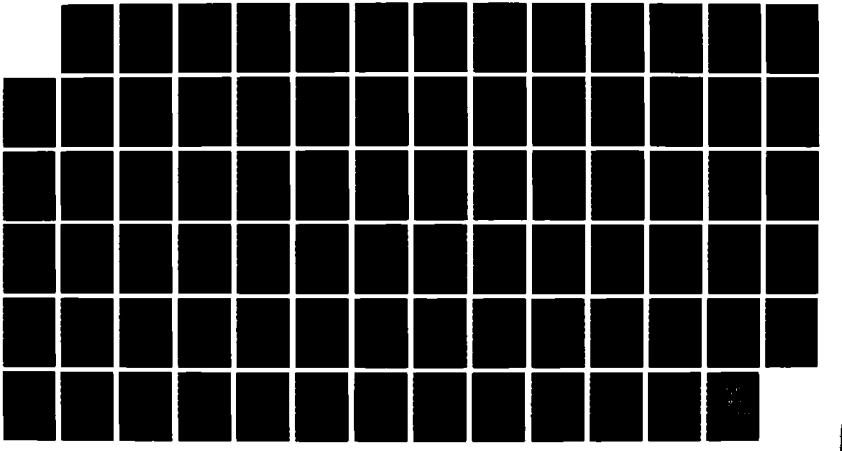
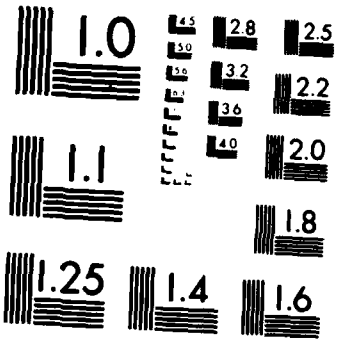




observations have been made by Hanes and Atlas (Reference 20). They showed very little loss of hydrocarbons, including the more volatile components, from oil-contaminated sediment placed in plexiglass trays on the sediment in Beaufort Sea, Alaska. Despite the inherent problems of sampling the contaminated sediment at the field site, of dilution with uncontaminated sediments and of the necessity to measure changes in hydrocarbons concentrations by ratiocination to a conservative tracer (tetradecane), we believe the mere detection of these hydrocarbons in samples taken during the experimental period signifies persistence, since there is no other obvious source of hydrocarbons. We detected no hydrocarbons in surface slicks or the water column at any time during the study, so it is unlikely these sources were causing recontamination of sediment by mixing within the pond or during sampling of the sediment. Uncontaminated sediments showed no peaks in the gas chromatographic analysis which might have been misconstrued as hydrocarbon peaks. Also, all sampling equipment and analytical glassware were thoroughly cleaned to prevent any cross-contamination of the environmental samples.

The observed persistence was probably the result of reduced volatility and lack of biodegradation. The lack of biodegradation can best be illustrated with the normal alkane, decane. In both deep and shallow water bottle tests, it was clear that faster biodegradation of decane relative to tetradecane was occurring, as evidenced by large differences in the ratios in sterile and non-sterile systems. In fact, examination of the actual hydrocarbon concentrations shows that both decane and tetradecane were degrading. In the field samples, however, decane to tetradecane ratios changed very little over time; examination of the actual concentrations of the hydrocarbons showed that it was not because both were degrading at equal ratios but it was because both hydrocarbons concentrations were relatively stable. Thus, biodegradation did not appear to be occurring in the field samples.

Oxygen limitation may have been responsible for this result; in fact, slightly greater loss of decane relative to tetradecane may have occurred in the shallow water site than in the deep water site. Based on our monitoring, we would expect more dissolved oxygen in the shallow water site. However, in the deep water bottle test, where dissolved oxygen concentrations were purposefully reduced by sparging the water with $N_2$ gas prior to test initiation, biodegradation of decane was evident. Thus, if oxygen was limiting biodegradation in the field, it was probably occurring at the sediment water interface. Limitation of biodegradation by available nitrogen and phosphorous concentrations seems unlikely in the field since the same water showed biodegradation of certain hydrocarbons (e.g., decane) in the bottle tests.

Similar conclusions could also be drawn by examining most of the other normal alkanes. The only other hydrocarbons to show possible biodegradation in the shallow water (1,2,5-TMB, indan, naphthalene) and deep water (naphthalene, methylhexane and the methylheptanes) bottle tests, all eventually disappeared in the field samples. Whether this was due to biodegradation could not be ascertained.

For those hydrocarbons which slowly disappeared in the field samples, it is difficult to delineate whether it was due to evaporation, biodegradation or dilution into the organic matrix of the sediment. Literature information supports the idea that, if the alkanes do not degrade, then many of the

other hydrocarbons will also not degrade. Biodegradation might be eliminated as a posssibility because of the persistence of the n-alkanes in the field sample. Dilution into the organic matrix of the sediment was probably occurring to some extent, since it would appear that low concentrations of residual hydrocarbons in the plexiglass trays did not eventually disappear. This could have been caused by the tray bottom preventing contact with, and dilution into, uncontaminated sediment.

Since many of the hydrocarbons in the field samples persisted longer at the deep water site than in the shallow water site, it is tempting to conclude that the greater water depth slowed losses to volatilization. Dilution into the organic matrix of uncontaminated sediment would not be expected to be affected by site differences (assuming equal distribution of organ carbon from site to site), thus, this loss mechanism can be down played. Biodegradation, as we indicated above, would probably not account for these differences, volatilization would appear to be the principal loss mechanism. Results from the bottle tests and the trays tend to support this conclusion; i.e., volatility appeared to be slightly less where larger volumes of water covered the contaminated sediment.

Results from the bottle tests and the plexiglass trays were not good predictors of events in the field. Bottle test data suggested that the majority of the hydrocarbons monitored in the jet fuel should evaporate within a few days. This was obviously not the case in the field, as seen by comparing Tables 1 and 2. On the other hand, bottles test data did suggest that 1,3,5-TMB, indan, naphthalene, and the higher molecular weight normal alkanes would evaporate slowly; these hydrocarbons, in fact, were also some of the slowest to disappear in the field samples. As we indicated above, the bottle tests further showed a potential for biodegradation of certain hydrocarbons, but a similar response could not be seen in the field samples. More rapid loss of hydrocarbons in the shallow water site of the pond, as we compared with the deep water site, was quite pronounced with some hydrocarbons. The bottle test results would not have clearly predicted this result. Although slight differences in the volatility of certain hydrocarbons were noted in shallow water and deep water bottle tests, it was apparent that larger bottles, which would allow greater water volumes to cover the contaminated sediment, are needed to properly simulate the deep water effect in the field.

Results from the plexiglass trays suggested that low concentrations of certain hydrocarbons should persist for greater than 20 days; clearly these hydrocarbons dropped below detectable limits in considerably less time in the field. If we assume volatility of the jet fuel hydrocarbons was equally probable from sediments in the pond and sediments in the plexiglass trays, then the long-term persistence of hydrocarbons in the trays probably reflected the isolation of contaminated sediments from uncontaminated sediments. Without prior knowledge of the importance of this limitation, however, estimates of hydrocarbon persistence from tray studies alone would be contradictory to actual events in the field. However, the tray data did suggest a lack of biodegradation (i.e., decane ratios with tetradecane remained relatively constant) and a greater loss of some hydrocarbons from the shallow water sites, both observations which are typical of the field data.

## SECTION III

## MISSILE FUEL STUDIES

### A. BACKGROUND

The active development of air-, sea-, and ground-launched missile systems has become one of the primary strategic and tactical weapons programs of the U.S. Air Force and U.S. Navy. A new era of fuel technology evolved when the Navy selected a specifically synthesized liquid hydrocarbon fuel, called RJ-5, to be used in the Talos missile (Reference 42). Since that time, the Air Force and Navy have continued to develop synthetic fuels for either turbine- or ram-jet powered missiles. The primary fuel evolved by the Air Force and Navy high-energy missile fuel technology was JP-9, a unique blend of liquid hydrocarbons which had the necessary high volatility, low freeze point, and high density. Missile fuels are composed of unique bridged-ring hydrocarbon structures containing high carbon-to-hydrogen ratios (Figure 62). Norbornane (bicyclo [2.2.1] heptane) is a basic part of each molecule, but is never, itself, present in their production or composition. These dienes are formed by the Diehls-Alder cycloaddition reaction (References 42 and 43). The diene constituents of RJ-5 and JP-9 are the norbornadiene and cyclopentadiene dimers, respectively. RJ-5, a completely synthetic fuel, is a mixture of three hexacyclic dihydro-di-(norbornadiene) components whose composition is shown in Table 3. JP-9 is a more volatile mixture, composed of cyclopentadiene and norbornadiene dimers along with several minor hydrocarbon components (Table 3) to increase the volatility and lower viscosity. The physical properties of these fuels are compared in Table 4. Both JP-9 and RJ-5 sorb to organic sediment more than petroleum-derived fuels such as JP-4 (reference 2).

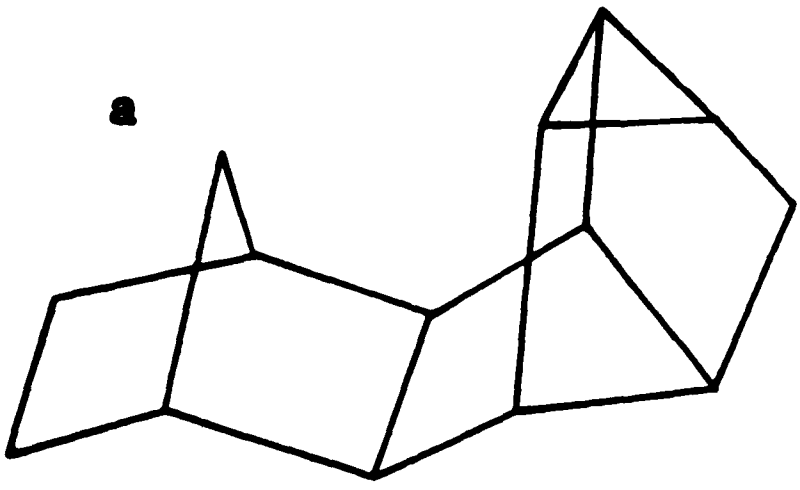
With increased usage, the potential for spillage and accidental release into the environment also increases. To predict the environmental impact of missile fuels, information on the fate of their constituent compounds as determined by solubility, volatility, sediment sorption and biodegradation must be obtained.

Little information is currently available on the potential environmental impact of the missile fuels in the environment. It was, therefore, important to study and assess the fate and toxicity of the fuels in aquatic systems. We undertook an examination of the fate and effects of missile fuels RJ-5 and JP-9 in aquatic environments using standard laboratory test procedures. The goal of this research was to determine the potential for missile fuel biodegradation in aquatic systems and the possibility of toxicity to microbial populations and aquatic invertebrates.

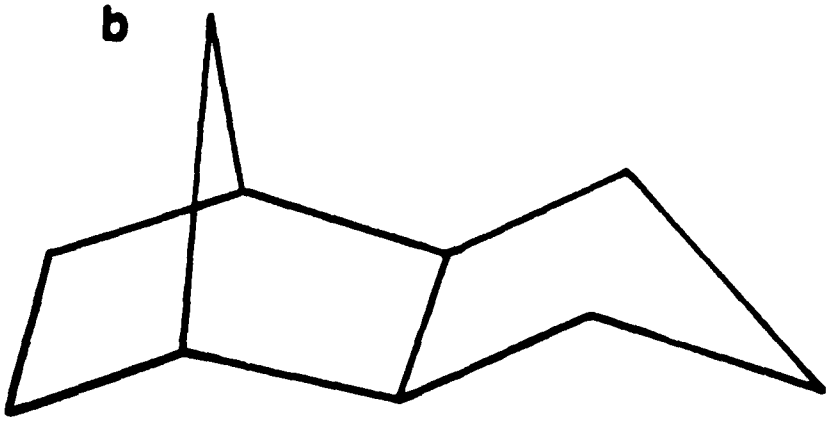
### B. METHODS

#### 1. Sampling

Water and sediment samples were collected at three sites near Pensacola, Florida; Bayou Chico, Escambia River, and Range Point Salt Marsh (Figure 2). The locations were selected to provide a range of salinities as well as a comparison between pristine and developed areas. Bayou Chico is located on the northern shore of Pensacola Bay in an industrial area, salinity ranges from 12 to 20 parts




endo, endo - DIHYDRODI (NORBORNADIENE)




exo - TETRAHYDRODI (CYCLOPENTADIENE)

Figure 62. Structure of Norbornadiene (A) and Cyclopentadiene (B) Dimers.

TABLE 3. MAJOR COMPONENTS OF RJ-5 AND JP-9[a]

| RJ-5 | Percent by Weight |
|---|---|
| Dehydro-Hexacyclic endo-endo-dihydrodi(norbornadiene) | 1.12 |
| Hexacyclic exo-endo-dihydrodi(norbornadiene) | 2.03 |
| Hexacyclic endo-endo-dihydrodi(norbornadiene) | 96.32 |
| **JP-9** | |
| N-Heptane | 1.0 |
| Methylcyclohexane | 7.1 |
| 2,5-Dimethylhexane | 0.8 |
| Toluene | 0.6 |
| exo-Tetrahydrodi(cyclopentadiene) | 66.8 |
| endo-Tetrahydrodi(cyclopentadiene) | 1.5 |
| endo, endo-Dihydrodi(norbornadiene) | 20.1 |

[a] Data taken from Smith et al. (Reference 43)

TABLE 4. TYPICAL PROPERTIES OF MISSILE AND AIRCRAFT FUELS[a]

| | JET FUELS | | MISSILE FUELS | | | |
|---|---|---|---|---|---|---|
| | JP-4 | JP-4 | JP-9 | JP-10 | RJ-4 | RJ-5 |
| Heating value, $MJ/m^3$ (Btu/gal) | 32,900 (118,000) | 34,800 (125,000) | 39,600 (142,000) | 39,600 (142,000) | 39,000 (140,000) | 44,900 (161,000) |
| Viscosity, $mm/^2$ at 233K(-40°F) | 4.5 | 17 | 24 | 19 | 60 | 2,000 |
| Freezing point, K (°F) | <215 (< -72) | <227 (< -51) | <219 (<-65) | <194 (<-110) | <233 (<-40) | >255 (>0) |
| Flashpoint, K (°F) | 244 (-20) | 388 (150) | 294 (70) | 327 (130) | 338 (150) | 383 (230) |
| Specific gravity at 289 K (60°F) | 0.77 | 0.83 | 0.94 | 0.94 | 0.94 | 1.08 |

[a]Data taken from Burdette et al., (reference 42)

per thousand, with hydrocarbon input from nearby industry and marinas. The Escambia River site is 8 miles upstream from the mouth of the Escambia River, north of local industry. Salinity is zero and the surrounding area is largely undeveloped. Range Point Salt Marsh is located on the north side of Santa Rosa Island, approximately 3 miles east of the Bob Sikes Bridge. Salinity varies from 10 to 20 parts per thousand and the area is nearly pristine.

Water was collected from each site by dip sampling with a clean glass bottle, transported to the laboratory, filtered through a 3-micron membrane filter, and stirred overnight at room temperature. The top 3 to 5 cm of sediment and associated detritus were collected at each site along with overlying water. The suspension was passed through a 2 mm screen, and particles of sand were allowed to settle. The resultant organic sediment slurry was decanted and stirred overnight at room temperature.

2. Fate Tests

Quiescent fate tests consisted of sets of four test flasks: (1) Active Water (AW) contained filtered water from the sampling site; (2) Sterile Water (SW) contained filtered water sterilized with 0.3 percent $HgCl_2$; (3) Active Sediment (AS) contained sediment (5-10 g/ℓ dry weight) and filtered water; (4) Sterile Sediment (SS) contained sediment, 0.3 percent Hg $Cl_2$, and filtered water from the test site. Quiescent tests were performed in 150 mℓ milk dilution bottles; duplicate bottles were prepared for each sample time. The final volume of liquid in each bottle was 25 mℓ. The missile fuel (10 µℓ) was added to the surface of the water in each bottle with a Drummond microdispenser, and the bottles were capped and shaken in a horizontal position for 10 minutes at 150 rpm to encourage initial sediment-fuel interaction. The caps were then removed and the bottles incubated as previously described for the jet fuel studies (Reference 2). An additional set of active and control bottles, used to monitor microbial populations and detect contamination of environmental samples, was prepared in the same manner. No hydrocarbons were added to the control bottles. After incubation, the total contents of each bottle were extracted with 10 ml of $CS_2$ that contained hexadecane as an internal standard. The extracts were placed in glass vials, sealed with silicone septa, and stored at -4 degrees centigrade until analyzed.

3. Chemical Analysis

Analysis was carried out by high-resolution capillary chromatography using a Hewlett Packard 5730 gas chromatograph equipped with flame ionization detector and cryogenic unit. The column was coated to a thickness of 1.0 um with methyl silicone bonded phase (Scientific Glass Engineering BP-1). Data integration and storage was performed by an HP3357 computer with Lab Automation System (LAS) capabilities. Inlet and detector temperatures were 250 degrees centigrade. Hydrogen was used as the carrier gas and nitrogen was used as make-up. Column flow was 1 L/minute. All samples were injected splitless. The temperature program for RJ-5 began at 90 degrees centigrade and increased 4 degrees centigrade/minute to 220 degrees centigrade. The temperature for JP-9 began at 25 degrees centigrade with a 4-minute isothermal hold and increased 4 degrees centigrade/minute to 200 degrees centigrade.

Statistical analyses of the data were performed using standard programs for means and standard deviations and simple linear regression. Regression analyses were performed on the semilog transformations of the concentration data.

4. Microbiology

Heterotrophic bacteria were enumerated by a standard five-tube most probable number (MPN) technique (Reference 44). The enumeration medium contained 1 gram of yeast extract and 5 g Bacto-Peptone (DIFCO) per liter. The pH was adjusted to 7.6 before autoclaving. Salinity values were matched to those at each sampling site with an aged sea salts solution. Each MPN tube was inoculated with 1.0 mℓ of an environmental sample. Tubes were incubated at 25 degrees centigrade temperature and examined for turbidity after 2 weeks.

Hydrocarbonoclastic bacteria were enumerated by a five-tube $^{14}C$-MPN technique similar to that described by Lehmicke et al. (Reference 45). The enumeration medium was a minimal salts broth (MSB) ($K_2HPO_4$, 700 mg; $MgSO_4 \cdot H_2O$, 11 2 mg; $ZnSO_4$, 5 mg; $NaMgSO_4 \cdot 2H_2O$, 2 mg; $CaCl_2$, 14 mg; $NH_4Cl$, 500 mg; one liter $H_2O$), adjusted to pH 7.6 and to the proper salinity with NaCL. One mL samples of the basal medium were dispensed into 4 mℓ Omnivials® (Wheaton Scientific). The vials were capped, sterilized by autoclaving, and stored at 5 degrees centigrade until used. The substrate was n-[1-$^{14}C$]-hexadecane (Amersham, Incorp.) with a specific activity of 235 µCi per mg. The hexadecane was diluted in hexane to a concentration of 8.25 µg/mℓ and 5.0 µℓ were transferred aseptically to sterile Sensi Discs® in a sterile petri dish. The hexane carrier was allowed to evaporate for 10 minutes, and the discs were distributed into the separate vials of MSB. The Sensi Discs® sank to the bottom of the vials, minimizing the volatilization of hexadecane from the medium. The substrate remained associated with the disc where it was available for degradation. This procedure resulted in a substrate concentration of 41 µg/ℓ and 20,000 dpm per vial. Each vial was inoculated with 0.1 mℓ of serial dilutions of a sample, and was incubated, without a cap, inside a tightly capped glass scintillation vial which contained 1 mℓ of 1 N NaOH (Figure 63). After incubation for 2 weeks the Omnivials® were removed, scintillation cocktail was added to the NaOH, and the radioactivity was measured by liquid scintillation counting in a liquid scintillation counter. Any vial that exceeded the background average by 1 percent or more of the total available counts was scored as positive.

5. Toxicity Assays

The bacteriostatic effect of RJ-5 was determined by measuring microbial activity in the presence of various concentrations of RJ-5.

Four exposure concentrations were tested (Control, 50 mg/ℓ, 500 mg/ℓ, 5000 mg/ℓ, at four times (4 hours, 24 hours, 48 hours, 72 hours). RJ-5 was dissolved in hexane and added to 1-liter flasks. The hexane was evaporated, and 1 liter of water and 500 mg sediment were added to each flask. The suspensions in the flasks were then homogenized with a polytron (Brinkman Inst. Co.) at full speed for 1 minute to emulsify the fuel. Control flasks received no fuel and were treated in the same manner as the exposed flasks. Triplicate flasks were prepared at each concentration. The fuel, sediment, and water mixtures were then transferred to 2-liter Erlenmeyer flasks and placed on a rotary incubator at 25°C and 100 rpm. At each exposure time, flasks were removed from the shaker, stirred on a magnetic stir plate, and sampled in 5 mℓ triplicates for measurement of activity. An

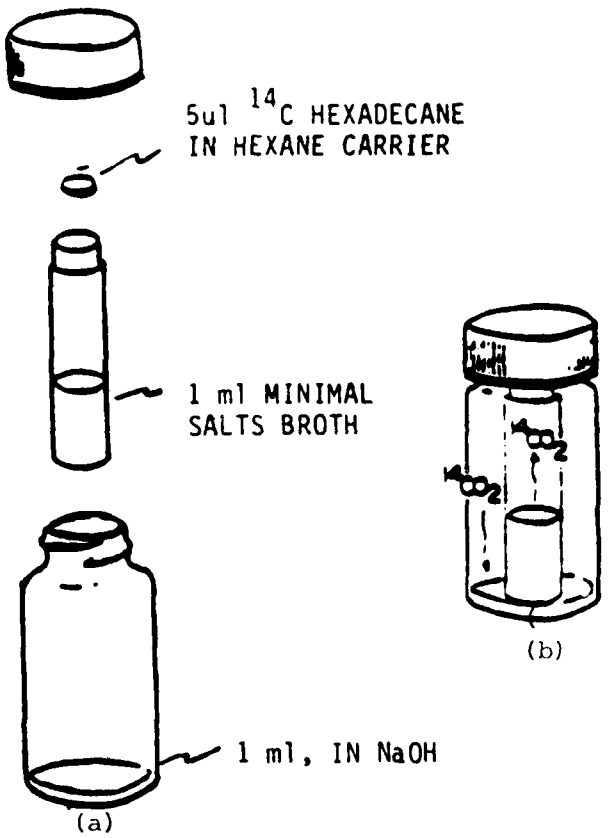



Figure 63. Enumeration of Hydrocarbon Degrading Microorganisms. a) Replicate Vials Received $^{14}$C-Hexadecane Sorbed onto Sterile Filter Discs, and 0.1 mL of Diluted Samples. b) Cultures were Incubated in Capped Scintillation Vials Containing 1 mℓ of 1N NaOH for 2 weeks.

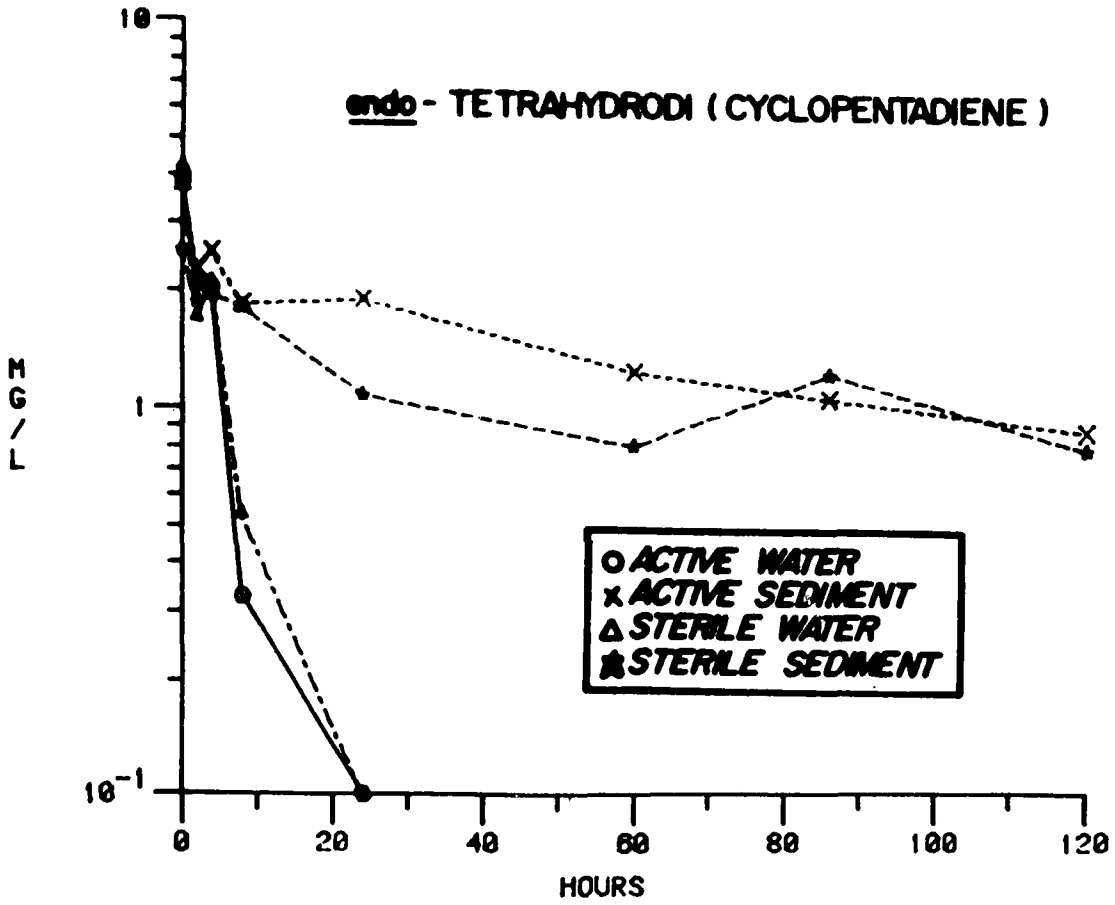



Figure 64. Fate of endo- Tetrahydrodi (Cyclopentadiene) of JP-9 in Sediment and Water from Range Point. Samples were Collected 11 October, 1983; Salinity was 21 Percent; Sediment Concentration in Experimental Bottles was 6.5 gram (Dry Weight)/liter. Data Shown are Means of Duplicate Analyses; Variation of Replicates was Less Than 10 Percent of Mean Values

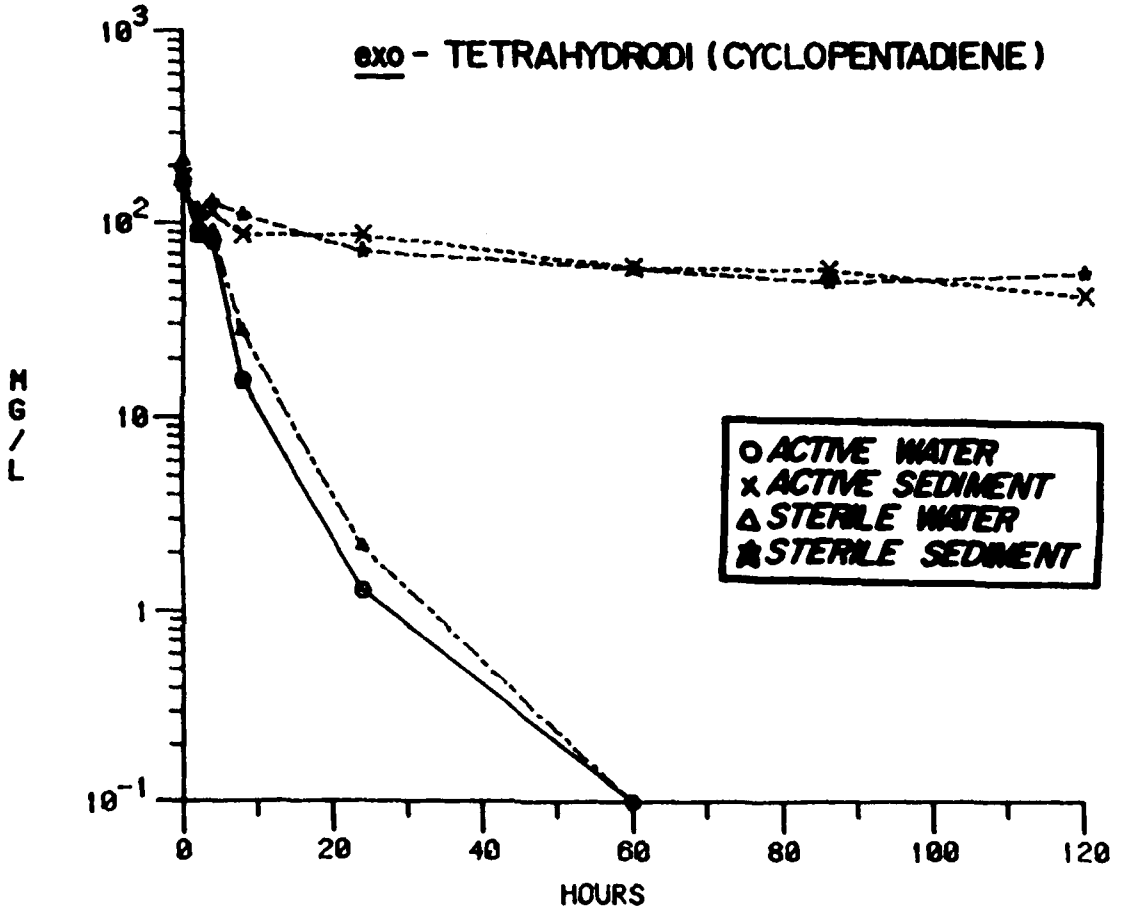



Figure 65. Fate of exo- Tetrahydrodi (Cyclopentadiene) of JP-9 in Sediment and Water from Range Point. Samples were Collected 11 October, 1983; Salinity was 21 Percent; Sediment Concentration in Experimental Bottles was 6.5 gram (Dry Weight)/liter. Data Shown are Means of Duplicate Analyses; Variation of Replicates was Less Than 10 Percent of Mean values.

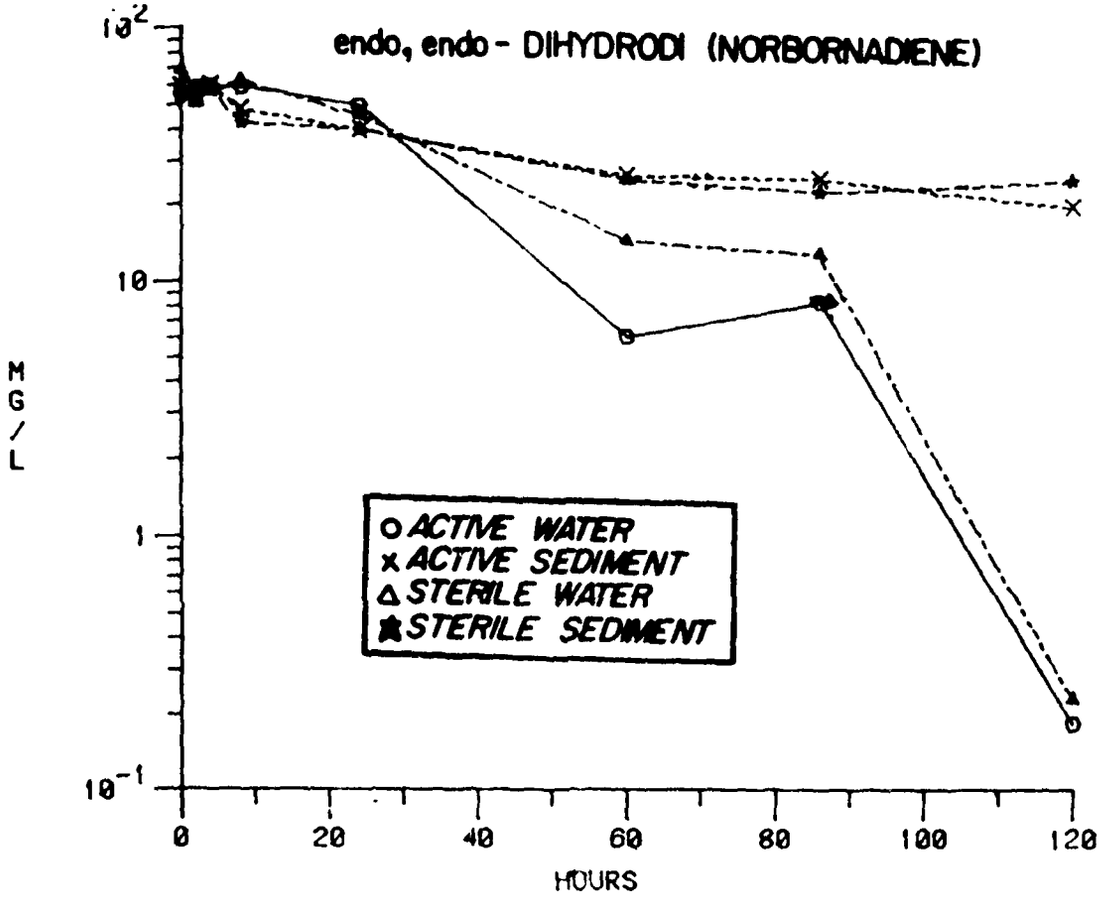



Figure 66. Fate of endo,endo- Dihydrodi (Norbornadiene) of JP-9 in Sediment and Water from Range Point. Samples were Collected 11 October, 1983; Salinity was 21 Percent; Sediment Concentration in Experimental Bottles was 6.5 gram (Dry Weight)/liter. Data Shown are Means of Duplicate Analyses; Variation of Replicates was Less Than 10 Percent of Mean Values.

additional sample was taken for microbial enumeration by acridine orange direct count (AODC).

Microbial activity was estimated by measuring glucose mineralization. Each 5 mℓ sample was placed in a 25 mℓ flask with 20 µg of $^{14}C$-labeled glucose/ℓ (Amersham Co.) incubated on a bench-top gyro rotary shaker at 125 rpm for 4-hours. At the end of the incubation period the medium was acidified to pH 3 and evolved $CO_2$ was trapped on filter paper saturated with 1ON NaOH. The amount of $^{14}CO_2$ trapped on the filter was quantitated by liquid scintillation counting and was used to determine the amount of glucose mineralized by the microbial community.

Toxicity to Mysidopsis bahia was measured in a standard 96-hour static test. Emulsions of the fuel were prepared as described above, except that sediment was omitted and samples were homogenized for 3 minutes. Mysids used in the tests were 48 to 72 hours old and were provided by Richard Montgomery, University of West Florida. LC-50 values were calculated by the Probit Method.

C. RESULTS

1. JP-9

When JP-9 was incubated with water from Range Point salt marsh, the fuel remained on the surface and the lighter components (n-heptane, methylcyclohexane, toluene and 2,5-dimethylhexane) volatilized within 5 hours (See Appendix C). Inclusion of sediment in the test system slowed the volatilization of methylcyclohexane and caused it to persist for 60 hours. There were no discernible differences between the active and sterile systems.

The high-density synthetic compounds disappeared more slowly (Figures 64 through 66 and Appendix C) because of their lower vapor pressures. The cyclopentadiene dimers were more volatile from water than the norbornadiene dimers. The endo dimers of cyclopentadiene were detectable in the water only at the initial sample time, while the exo dimers persisted for 24 hours. Endo, endo-dihydrodi(norbornadiene) volatilized more slowly from water and was still detectable after 120 hours. No appreciable losses of high-density components occurred from test systems that contained sediment. Sorption to sediment essentially precluded volatilization of both the norbornadiene dimers and the more volatile cyclopentadiene dimers. The results were identical in sterile controls, indicating that the sorption and volatilization processes were not confounded by biotic factors.

Results were similar when the experiment was repeated with sediment and water samples from the Escambia River (see Appendix C). Except for methylcyclohexane, the lighter components volatilized from water within 8 hours, but persisted for 24 hours in systems that contained sediment. Methylcyclohexane remained detectable in sediment throughout the 120-hour test. Cyclopentadiene dimers volatilized from water within 24 hours, whereas the norbornadiene dimer remained at a detectable, but greatly reduced concentration, through 120 hours. The high-density dimers did not disappear from test systems that contained sediment.

2. RJ-5

RJ-5 sank to the bottom of the water and sediment samples with no detectable biodegradation or volatilization of the norbornadiene dimers after 1400-hour incubation (Figure 67 through 69; Appendix C). Data shown are for samples from Range Point; results were identical with samples from Escambia River incubated for 2000 hours (see Appendix C).

3. Toxicity

Comparison of microbial population densities (Figure 70) in RJ-5 treated samples, with those in controls, indicated that RJ-5 was not toxic to the bacteria in the biodegradation test. Indeed, RJ-5 seemed to stimulate microbial populations, possibly because the traces of lighter hydrocarbon components served as sources of carbon and energy for the microorganisms. Similar results were obtained with JP-9.

The effects of RJ-5 emulsions on microbial activity were assessed by measurement of glucose mineralization (Figure 71). RJ-5 initially inhibited the heterotrophic activity of microbial communities. After 24 hours, the activity returned to control levels or above in suspensions with 50 or 500 mg RJ-5 per liter. Activity remained depressed in suspensions treated with 5000 mg/ℓ.

Because RJ-5 settles to the bottom of aquatic systems, experiments were designed to test its toxicity to the benthic invertebrate Mysidopsis bahia. Sediment was not included in the experimental systems because it would have made it difficult to count the mysids. The 96-hour LC-50 for fuel emulsified in water was 88 µg/ℓ (Table 5). Most of the mortality occurred within the first 24 hours of the test which suggests that traces of volatile compounds might have been responsible for the toxicity. Alternatively, the fuel-water emulsion may have separated during the prolonged incubation period and lessened the contact of the mysids with the fuel.

D. DISCUSSION

The differences in density between RJ-5 and JP-9 seem to determine the differences in their fate in aquatic systems. Volatilization of the components from JP-9 was much faster than from RJ-5 because JP-9 floats on the surface of the water. The difference was particularly notable with endo-endo-dihydrodi(norbornadiene), which was present in both fuels; it volatilized from JP-9 mixtures within a few days, yet remained for much longer periods in RJ-5. Sorption to the sediment greatly reduced the volatilization of both fuels. The norbornadiene dimers have a higher affinity for sediment than the cyclopentadiene dimers (Reference 46), but both types of compounds resisted weathering in the presence of sediment.

The structural features of the high-density synthetic molecules make them very resistant to microbial attack. Preliminary work by MacIntyre et al. (Reference 46) has suggested that some of the norbornadiene dimers can be biologically transformed by the insertion of an oxygen atom, but we did not detect such transformations.

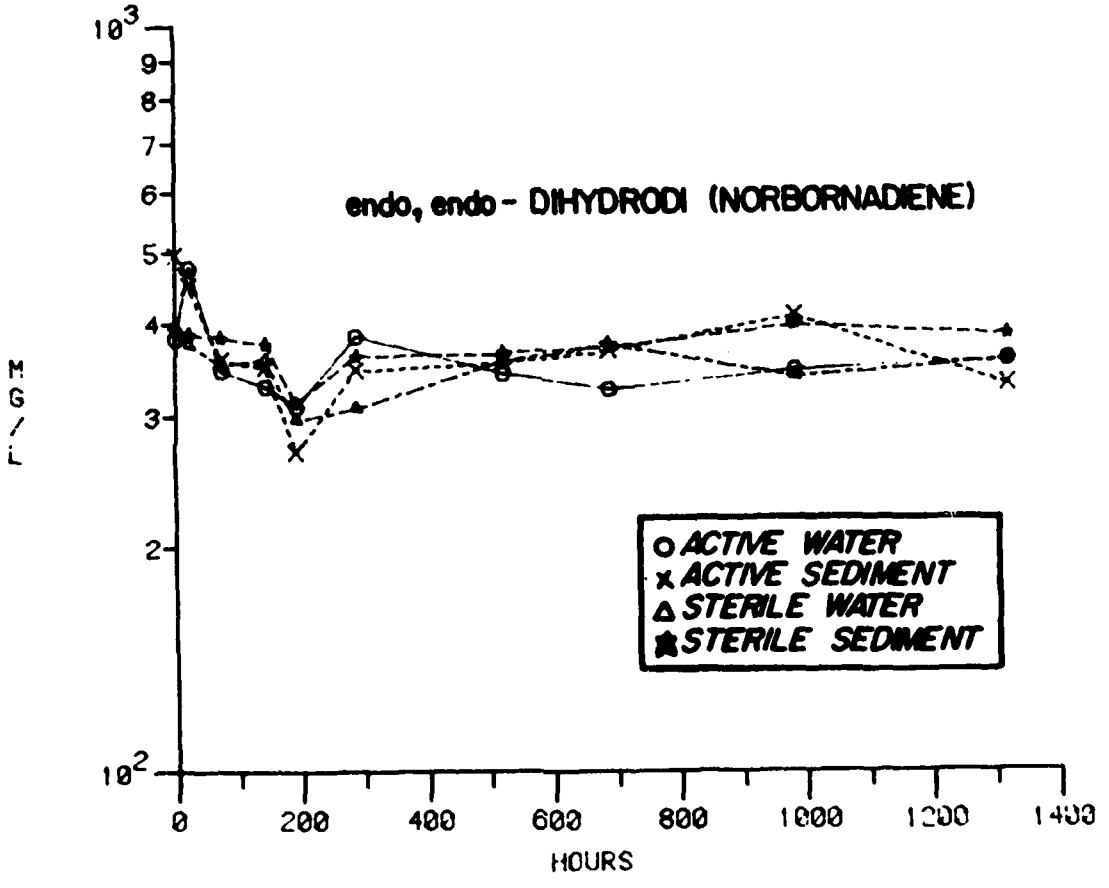



Figure 67. Fate of endo,endo- Dihydrodi (Norbornadiene) of RJ-5 in Sediment and Water from Range Point. Samples were Collected 6 April, 1983; Sediment Concentration in Experimental Bottles was 4.7 grams (Dry Weight)/liter. Data Shown are means of Duplicate Analyses; Variation Between Replicates was Less Than 10 Percent of Mean Values.

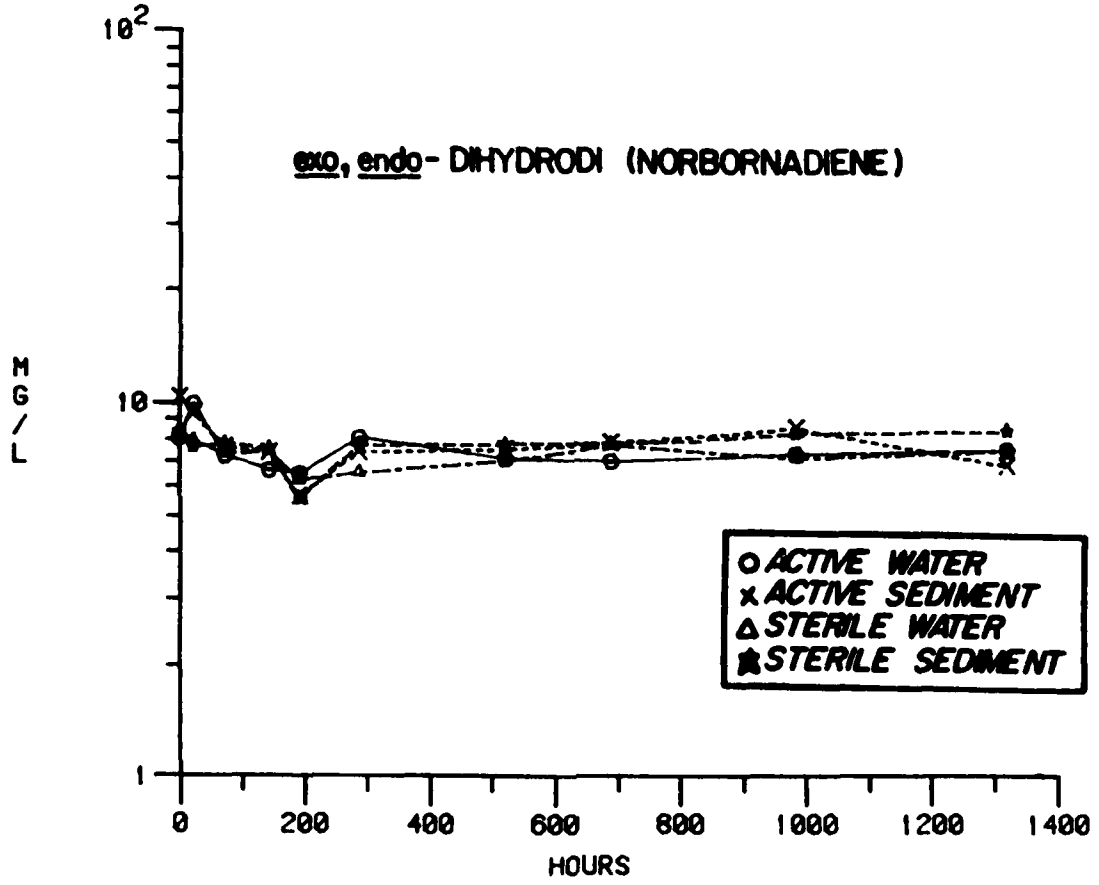



Figure 68. Fate of exo,endo- Dihydrodi (Norbornadiene) of RJ-5 in Sediment and Water from Range Point. Samples were Collected 6 April, 1983; Sediment Concentration in Experimental Bottles was 4.7 grams (Dry Weight)/liter. Data Shown are means of Duplicate Analyses; Variation Between Replicates was Less Than 10 Percent of Mean Values.

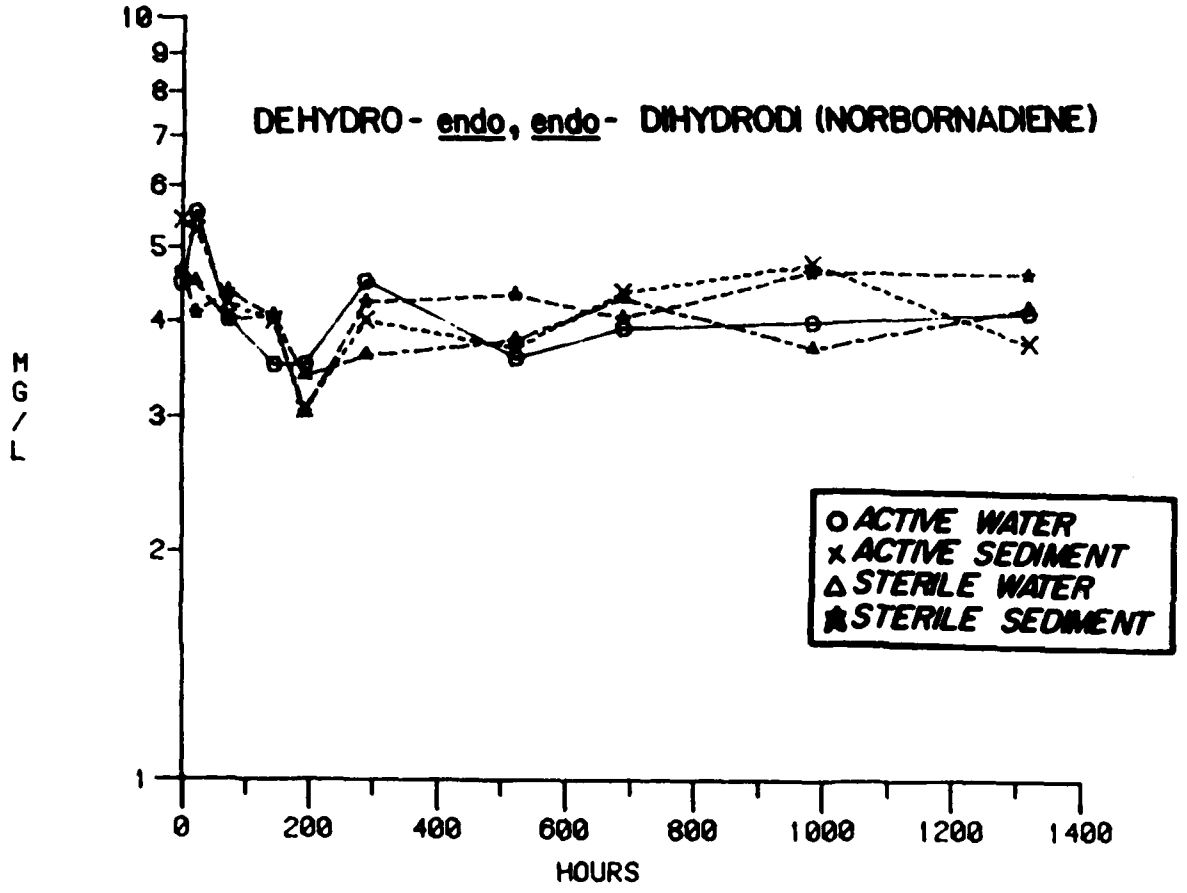



Figure 69. Fate of Dehydro- endo,endo- Dihydrodi (Norbornadiene) of RJ-5 in Sediment and Water from Range Point. Samples were Collected 6 April, 1983; Sediment Concentration in Experimental Bottles was 4.7 grams (Dry Weight)/liter. Data Shown are means of Duplicate Analyses; Variation Between Replicates was Less Than 10 Percent of Mean Values.

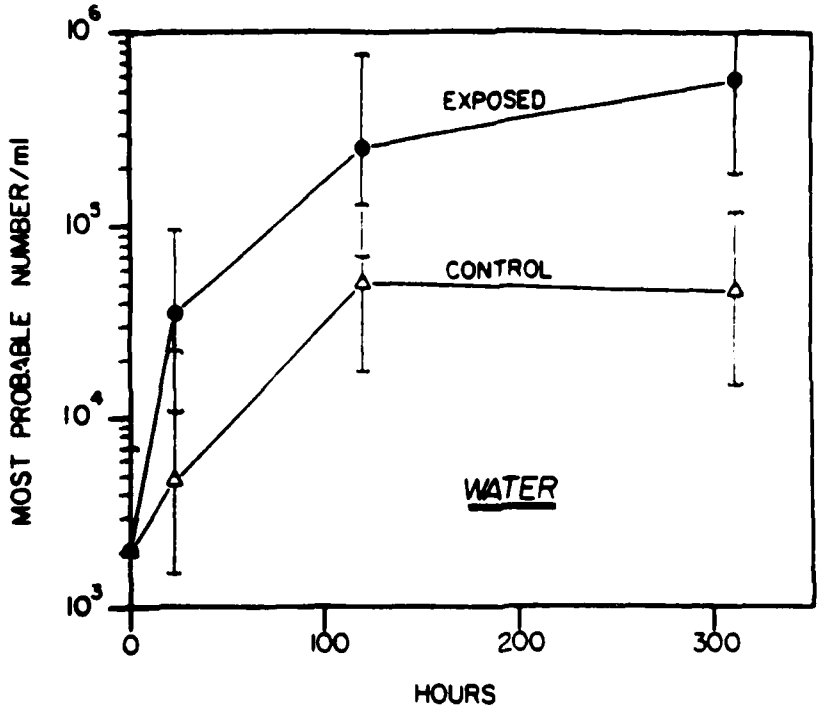



Figure 70. Effects on RJ-5 on Microbial Community Size in Water from Range Point. Water was Incubated with RJ-5 at a Concentration of 400 µg/liter as Described in Methods. Samples were Removed at Appropriate Intervals and Bacteria were Enumerated by Measurements of MPN in Nutrient Broth.

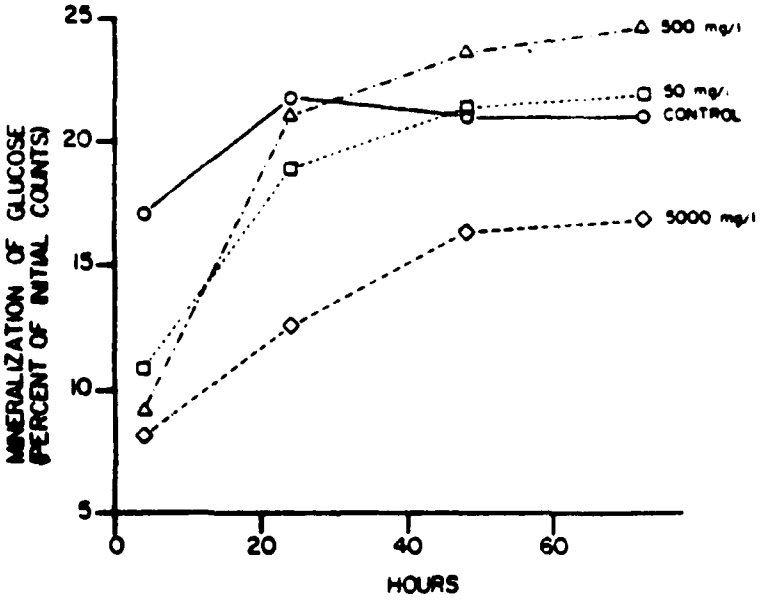



Figure 71. Effects of RJ-5 on Microbial Activity. Glucose Mineralization was Measured in Range Point Sediment and Water Samples after Treatment with Various Concentrations of RJ-5 as Described in Methods.

TABLE 5. TOXICITY OF RJ-5 TO MYSIDOPSIS BAHIA.

| Concentration (µg/ℓ) | Percent Mortality (96h) |
|---|---|
| Control | 15 |
| 62 | 30 |
| 96 | 70 |
| 148 | 80 |
| 227 | 100 |
| 349 | 100 |
| LC50[a] | 87.6 ± 16.7 µg/ℓ |

[a] Probit analysis with Abbott's correction for control mortality (N=20)

The fuels were not toxic to microorganisms at low to moderate concentrations but RJ-5 was toxic to mysid shrimp. The LC-50 values observed in this study were similar to those reported by Jenkins et al. (Reference 47) for flagfish and rainbow trout. Because the high-density components of the fuels are essentially insoluble in water, the figures given for nominal concentrations in the microbial toxicity tests should be considered to reflect fuel-water ratios, rather than concentrations. The lower concentrations in the mysid toxicity tests are closer to the solubility of the fuel components.

The recalcitrance of RJ-5 to microbial degradation, affinity for sediment, density, and toxicity to aquatic macrobiota suggest that a large-scale spill of the fuel could present major environmental problems. JP-9 appears to be much less of a potential problem in the aquatic environment than RJ-5 because of its higher vapor pressure and lower density.

# SECTION IV

# CONCLUSIONS AND RECOMMENDATIONS

## A. CONCLUSIONS

The following conclusions are presented, based on laboratory and field research over the past year.

1. If jet fuel is spilled on the surface of a quiescent body of water, it will rapidly volatilize into the atmosphere. Very little, of the hydrocarbons will dissolve into the water column and persist therein. Based on observations during the field study, jet fuel that had reached the water surface during dosing rapidly evaporated. In addition, sampling and extraction of large water volumes and gas chromatographic analysis of the extracts indicated no hydrocarbons in the water 2 days following the dosing.

2. Mechanical mixing of the jet fuel hydrocarbons within the water column is required before hydrocarbons will be retained and detected therein. This was concluded largely from our previous quiescent bottle tests; initial shaking of the fuel oil with the water for 1 hour before incubation under quiescent conditions resulted in detectable concentrations of hydrocarbons in the water. These hydrocarbon concentrations then decreased steadily by volatilization over time, reaching undetectable levels in approximately 2-18 days, depending on water source and the hydrocarbon. Unfortunately, this mixing step could not be accommodated in the field study, therefore, field validation of these results was not possible.

3. Dosing of the field site with jet fuel-contaminated sediments proved to be both practical and efficient for examining the fate of hydrocarbons in sediments. Our method modeled an extreme situation; i.e., extensive mechanical mixing of spilled fuel with water and suspended sediments. Both the laboratory and field experiments showed this situation will result in long-term (greater than 30 days) association of hydrocarbons with sediments.

4. Volatilization of sediment-sorbed hydrocarbons was slow after the contaminated sediments had settled to the bottom of the associated pond or water body. Concentrations of many of the hydrocarbons associated with the sediments slowly decreased, with detectable concentrations remaining for 15 to 20 days. Greater depth of the water column and possible reduced oxygen conditions increased the persistence.

5. Dilution of sediment-sorbed hydrocarbons into the organic matrix of surrounding uncontaminated sediments probably occurred. This conclusion is based on the observation that concentrations of most hydrocarbons associated with unconfined sediments (i.e., not in field trays) fell below detection limits in the field despite the apparent absence of volatility and biodegradation. Only the high molecular weight n-alkanes were an exception, they were consistently found in all sediment samples during the entire experimental period. In addition, concentrations of the methylsubstituted alkanes, ethylcyclohexane, p-xylene and 1,3,5-trimethylbenzene did not substantially decrease at the deeper more anaerobic site.

6. Biodegradation was not a major factor in the loss of hydrocarbons from sediments in our test pond as several hydrocarbons which readily biodegraded in the bottle tests, did not disappear at the field site. Possibly, this was the result of a more anaerobic environment associated with the sediment bed in the field, a condition which is known to reduce biodegradation rates.

7. The information produced from the quiescent bottle tests, as they have been used in this project to study the fate of JP-4 in sediments, accurately forecast events in the field in some instances but disagreed with results from the field in other cases. For example, most of the hydrocarbons that persisted in the bottle tests also persisted in the field. However, most of the hydrocarbons that appeared to be biodegraded in the bottle tests did not disappear from the plexiglass trays and were slow to disappear in the field samples. In addition, many of the hydrocarbons that rapidly volatilized from the sediments in the bottle tests, were much slower to volatilize from sediments in the field. These differences were probably due to physical limitations of the bottle test inadequately representing the volume of water over the sediment in the field.

8. JP-9 and RJ-5 missile fuels were not biodegradable. Because of its higher density, RJ-5 will persist if spilled in aquatic systems. These fuels, particularly RJ 5 could be toxic to marine animals.

## 8. RECOMMENDATIONS

1. Assessments of jet fuel fate in aquatic systems should be carefully considered, particularly where suspended sediments may be involved.

2. Biodegradation potential should be carefully examined in laboratory and field tests. Experiments should be conducted in the laboratory under conditions in which degradation results are not confounded by volatilization. Biodegradation products of individual hydrocarbons should be determined to provide a more definitive method for assessing biodegradation in the laboratory and in the field.

3. The role of sediments in stimulating or suppressing biodegradation in aquatic systems should be more thoroughly studied.

4. More information is needed on the biodegradation of the water-soluble fractions of the jet fuels. This fraction, because of the solubility of the associated aromatic hydrocarbons, is most likely to move from the sediments into the water column. The persistence of some of these toxic aromatic hydrocarbons in the sediments, as observed in the field study, may mean that biodegradation in the water column is one of the few mechanisms for eventual elimination of the contamination.

5. Adaptation of microbial communities to faster biodegradation of hydrocarbons should be examined in field and laboratory systems since this may reveal a community response that is not reflected in the initital disappearance of parent compound.

6. The plexiglass trays, which were used in our study to conduct field experiments on the environmental significance of laboratory data, should be further developed as a test method. This should include additional field validation studies, improvements in sampling techniques, the possible use of sterilized sediments to

single out events due to biodegradation, and optimization of tray design relative to water exchange with the sediment surface and anaerobic conditions in the sediment. Addition of a conservative tracer, such as the hydrocarbon pristane (very slow to biodegrade or volatilize), to the jet fuel should be tested as a means of improving quantitative chemical analysis of field samples.

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# APPENDIX A

## DATA TABLES AND FIGURES FOR FIELD SITE CHARACTERIZATION

TABLE A-1. SALINITY MEASUREMENTS (PARTS PER THOUSAND) AT FIELD SITE

| DATE | B | C-SURFACE | C-MIDDLE | C-BOTTOM |
|---|---|---|---|---|
| 4-26 | ND[b] | 10 | 10 | 10 |
| 5-1 | ND | ND | ND | 8 |
| 5-7 | ND | 18 | 19 | 20 |
| 5-10 | ND | 18 | 18 | 18 |
| 5-16 | 17 | ND | 17 | ND |
| 5-18 | 15 | 16 | 17 | 17 |
| 5-21 | 17 | 16 | 17 | 19 |
| 5-25 | 15 | 15 | 17 | 19 |
| 6-3 | | DOSING | | |
| 6-11 | 15 | 16 | 18 | 20 |
| 6-18 | 20 | 20 | 21 | 22 |
| 6-25 | 18 | 18 | 20 | 21 |
| 7-10 | 14 | 14 | 20 | 20 |
| 7-17 | 16 | 17 | 22 | 22 |

[a] Samples taken at SITE C were approximately 15 cm (SURFACE), 175 cm (MIDDLE) and 350 cm (BOTTOM: SEDIMENT WATER INTERFACE) below the water surface.

[b] ND, not determined.

TABLE A-2. DISSOLVED OXYGEN MEASUREMENTS (mg/ℓ) AT THE FIELD SITE

| DATE | B | C-SURFACE | C-MIDDLE | C-BOTTOM |
|---|---|---|---|---|
| 5-1 | ND | 7.9 | 13 | .4 |
| 5-4 | ND | 6.4 | 5.0 | 3.5 |
| 5-7 | ND | 6.0 | 5.5 | 1.1 |
| 5-9 | ND | 7.1 | 6.3 | 1.2 |
| 5-10 | 7.4 | 7,3 | 6.6 | 1.5 |
| 5-14 | 6.5 | 6.2 | 1.2 | .8 |
| 5-16 | 7.5 | 7.5 | ND[b] | ND |
| 5-18 | ND | 5.9 | 3.3 | 2.8 |
| 5-21 | 4.6 | 4.5 | 2.3 | 1.8 |
| 5-25 | 5.2 | 5.1 | 2.9 | .7 |
| 6-1 | 6.7 | 6.7 | 5.8 | 5.1 |
| 6-3 | | DOSING | | |
| 6-5 | 5.7 | 5.6 | 4.0 | 2.1 |
| 6-6 | 6.2 | 5.9 | 4.9 | 1.0 |
| 6-8 | 6.9 | 7.1 | 5.2 | .8 |
| 6-11 | 4.2 | 4.9 | 2.4 | 2.8 |
| 6-14 | 6.7 | 7.2 | 5.5 | .7 |
| 6-18 | 5.6 | 5.5 | 5.0 | 4.9 |
| 6-25 | 5.1 | 5.3 | 2.4 | 1.1 |
| 7-2 | 6.1 | 6.0 | 4.9 | 2.6 |
| 7-10 | 5.3 | 4.9 | .4 | .2 |
| 7-12 | 5.0 | 5.0 | 4.2 | 4.3 |

[a] Samples taken at Site C, were approximately 15 cm (SURFACE), 175 cm (MIDDLE) and 350 cm (BOTTOM: SEDIMENT-WATER INTERFACE) below the water surface.
[b] ND, not determined.

TABLE A-3. MEASURED VALUES OF pH AT THE FIELD SITE

| Date | Sites[a] B | C |
|---|---|---|
| 4-26 | 7.5 | 7.5 |
| 4-27 | 7.2 | 7.4 |
| 5-1 | 7.0 | 7.1 |
| 5-7 | 7.6 | 7.6 |
| 5-9 | 7.6 | 7.6 |
| 6-3 | DOSING | |
| 6-5 | 7.1 | 6.9 |
| 6-6 | 6.9 | 7.1 |
| 6-7 | 7.3 | 7.2 |
| 6-8 | 6.7 | 7.0 |
| 6-11 | 6.6 | 6.8 |
| 6-14 | 7.2 | 6.6 |
| 6-18 | 8.4 | 7.9 |
| 6-25 | 6.4 | 6.8 |

[a] All samples taken approximately 15 cm below water surface.

TABLE A-4. MEASUREMENTS OF TEMPERATURE (°C) AT FIELD SITE SITES[a]

| DATE | B | C-SURFACE | C-MIDDLE | C-BOTTOM |
|---|---|---|---|---|
| 5-4 | ND[b] | 26.0 | 25.0 | 25.0 |
| 5-7 | ND | 29.5 | 28.8 | 28.5 |
| 5-9 | ND | 25.0 | 23.0 | 23.0 |
| 5-10 | 25.0 | 28.0 | 24.0 | 24.0 |
| 5-14 | 33.0 | 31.0 | 28.0 | 28.0 |
| 5-16 | 30.0 | 30.0 | 26.0 | 26.0 |
| 5-18 | - | 25.0 | 25.0 | 25.0 |
| 5-21 | 28.0 | 28.0 | 27.0 | 27.0 |
| 5-25 | 29.0 | 29.0 | 29.0 | 29.0 |
| 6-1 | 29.0 | 28.0 | 24.0 | 24.0 |
| 6-3 | | DOSING | | |
| 6-5 | 32.0 | 33.0 | 27.0 | 27.0 |
| 6-6 | 29.0 | 29.0 | 27.0 | 28.0 |
| 6-7 | 33.0 | 34.0 | 33.0 | 28.0 |
| 6-8 | 34.0 | 34.0 | 29.0 | 20.0 |
| 6-11 | 31.0 | 33.0 | 29.0 | 29.0 |
| 6-14 | 29.0 | 30.0 | 27.0 | 29.0 |
| 6-18 | 30.0 | 30.0 | 30.0 | 30.0 |
| 6-25 | 30.0 | 30.0 | 31.0 | 32.0 |
| 7-? | 30.0 | 29.0 | 31.0 | 32.0 |
| 7-10 | 36.0 | 36.0 | 33.0 | 33.0 |
| 7-17 | 32.0 | 32.0 | 32.0 | 31.0 |

[a] Samples taken at Site C were approximately 15 cm (SURFACE), 175 cm (MIDDLE) and 350 cm (BOTTOM: SEDIMENT-WATER INTERFACE).
[b] ND, not determined.

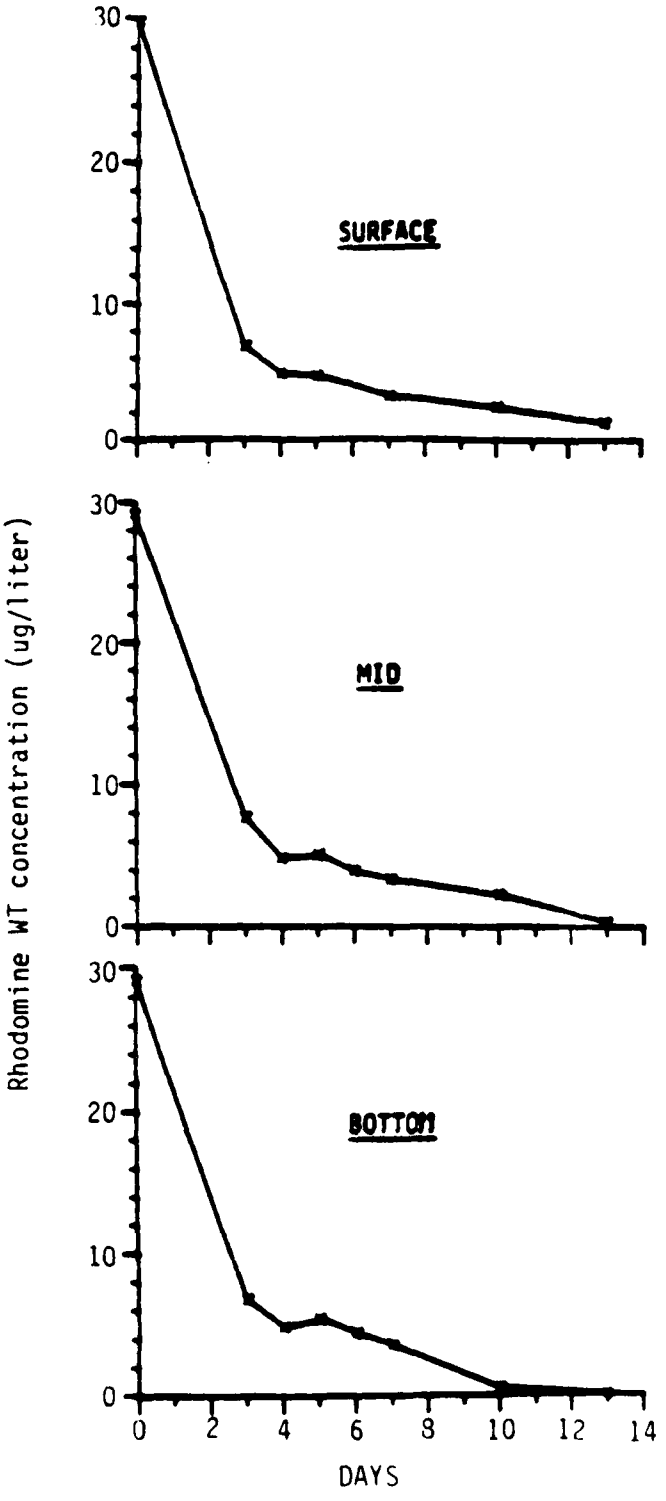


Figure A-1. Rhodomine WT Concentrations at Site C in the Test Pond at Approximately 15 cm (Surface), 175 cm (Middle), and 350 cm (Bottom: Sediment Water Interface Below the Water Surface.

# APPENDIX B

## DATA TABLES (JET FUEL FIELD STUDY)

TABLE B-1. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER ACTIVE BOTTLES TESTS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| BENZENE | 2.6 | 0 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| HEPTANE | 6.9 | 0 | 0 | 0 | 0 | 0 |
| METHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE | 5.8(5.2) | 5.2(0.3) | 1.6 | 6.3(0.3) | 2.28 | 4.1(3.4) |
| 2-METHYLHEPTANE | 2.0(0.8) | 3.2(2.2) | 3.5 | 0 | 0 | 0 |
| 3-METHYLHEPTANE | 2.5(1.0) | 3.2(1.3) | 4.6 | 2.0(0.1) | 1.8(0.5) | 0 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| OCTANE | 5.8(2.4) | 6.0(2.7) | 4.9 | 0 | 0 | 0 |
| ETHYLCYCLOHEXANE | 2.1(0.9) | 2.5(1.0) | 3.4 | 1.5(0.1) | 1.5(0.1) | 0 |
| ETHYLBENZENE | 0 | 0 | 0 | 0 | 0 | 0 |
| M-XYLENE | 1.16 | 0 | 0 | 0 | 0 | 0 |
| P-XYLENE | 1.59 | 1.2(0.4) | 1.7 | 0 | 1.1(0.2) | 0 |
| O-XYLENE | 0 | 0 | 0 | 0 | 0 | 0 |
| NONANE | 11(4.3) | 11(3.5) | 9.7 | 3.1 | 2.4(0.4) | 0 |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 3.5(1.4) | 3.7(1.0) | 4.3 | 2.6(0.2) | 2.9(0.5) | 1.5 |
| 1,2,4-TRIMETHYLBENZENE | 11(1.1) | 9.0(1.7) | 11 | 8.9(0.4) | 4.6(5.7) | 5.4 |

TABLE B-1. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER ACTIVE BOTTLES TESTS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| DECANE | 15(2.6) | 20(3.5) | 20 | 7.3(1.6) | 6.3(1.5) | 1.4 |
| 1,2,3-TRIMETHYLBENZENE | 1.8 | 0 | 0 | 0 | 0 | 0 |
| INDAN | 1.3(0.1) | 1.2(0.3) | 0 | 0 | 0 | 0 |
| UNDECANE | 0 | 0 | 1.1 | 1.4(0.1) | 15(3.6) | 4.5(2.9) |
| NAPHTHALENE | 7.9(1.5) | 7.9(0.8) | 7.9 | 4.7(0.1) | 4.8(0.3) | 2.2(0.9) |
| DODECANE | 46(5.5) | 51(2.9) | 49 | 33(0.7) | 30(3.3) | 15(5.5) |
| TRIDECANE | 30(3.0) | 46(3.3) | 42 | 33(1.3) | 30(2.9) | 21(6.1) |
| TETRADECANE | 21(1.3) | 26(1.3) | 23 | 20(0.9) | 19(1.8) | 15(2.9) |
| PENTADECANE | 6.7(0.4) | 80(0.4) | 7.1 | 6.2(0.2) | 6.1(0.6) | 5.4(0.9) |
| HEXADECANE | 0.7 | 0.6(0.1) | 0.5 | 0.6(0.1) | 1.8(1.9) | 0.4 |

[a] Values are means (± the standard deviations). Where no standard deviation is indicated, n=1; i.e., hydrocarbon concentrations in the other samples was below detection limits. Only one bottle was analyzed for Day 4 samples.

[b] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-2. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER STERILE BOTTLE TESTS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| BENZENE | 5.6(0.9) | 0 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 0 | 0 | 0 | 1.3 | 0 | 0 |
| HEPTANE | 0 | 0 | 8.9 | 28 | 38 | 0 |
| METHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE | 0 | 5.1(0.5) | 3.6(0.1) | 4.6(2.2) | 2.4 | 2.7(0.2) |
| 2-METHYLHEPTANE | 2.0 | 1.8(0.2) | 3.1(1.1) | 9.4 | 1.3 | 1.1 |
| 3-METHYLHEPTANE | 2.1(0.7) | 2.4(0.3) | 2.9(1.6) | 6.6(7.2) | 1.9 | 1.5 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| OCTANE | 4.3(1.6) | 4.4(2.1) | 7.4(1.2) | 14(16) | 3.3(1) | 3.0(0.8) |
| ETHYLCYCLOHEXANE | 1.8(0.6) | 1.9(0.2) | 2.9(1.0) | 7.9 | | 1.1 |
| ETHYLBENZENE | 0 | 0 | 0 | 0.9 | 0 | 0 |
| M-XYLENE | 0 | 0 | 0 | 0 | 0 | 0 |
| P-XYLENE | 0 | .95(0.1) | 1.6(0.4) | 3.9 | 0.93 | 0.94 |
| O-XYLENE | 0 | 47(0.5) | 48 | 37 | 1.8(2.6) | 0 |
| NONANE | 8.8(3.4) | 8.5(3.4) | 13(0.4) | 23(22) | 9.5(2.0) | 10(1.5) |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 2.8(1.1) | 2.3(1.0) | 3.9(0.6) | 4.9(4.7) | 2.2(0.3) | 2.3(0.3) |
| 1,2,4-TRIMETHYLBENZENE | 8.9(0.6) | 7.2(0.3) | 9.5(0.3) | 19 | 4.7(5.1) | 8.2(1.2) |

TABLE B-2. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER STERILE BOTTLE TESTS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| DECANE | 18(5.9) | 17(6.2) | 22(6.3) | 32(27) | 18(1.8) | 20(1.9) |
| 1,2,3-TRIMETHYLBENZENE | 0 | 0 | 0 | 0 | 0 | 0 |
| INDAN | 0 | .8(.06) | 1.1(0.4) | 2.0 | 1.2 | 1.0 |
| UNDECANE | 34(8.3) | 32(12) | 38(17) | 52(37) | 34(30) | 36(2.1) |
| NAPHTHALENE | 7.7(1.9) | 6.5(22) | 8.0(2.5) | 7.8(5.5) | 5.6(0.3) | 6.0(0.7) |
| DODECANE | 45(9.3) | 42(13) | 49(23) | 59(37) | 43(3.2) | 44(1.3) |
| TRIDECANE | 39(9.0) | 36(12) | 41(19) | 49(29) | 41(2.5) | 39(0.1) |
| TETRADECANE | 22(3.9) | 21(6.4) | 24(13) | 29(16) | 23.9(1.4) | 23.0(1.7) |
| PENTADECANE | 7.1(1.3) | 7.1(2.2) | 7.3(4.0) | 8.9(5.1) | 7.9(0.6) | 7.2(0.6) |
| HEXADECANE | .5(0.1) | 0.7(0.1) | .08 | 0.9(0.3) | 0.5 | 0.6(0.1) |

[a] Values are means (± the standard deviations). Where no standard deviation is indicated, n=1; i.e., hydrocarbon concentrations in the other samples was below detection limits.

[b] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-3. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER ACTIVE BOTTLE TESTS.

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| BENZENE | 11 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| HEPTANE | 13(1.5) | 6.8(3.8) | 11 | 0 | 0 |
| METHYLCYCLOHEXANE | 1.8(0.1) | 1.1(.02) | 1.3 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE | 1.5 | 4.1(0.7) | 4.6 | 2.9(0.5) | 2.4(0.2) |
| 2-METHYLHEPTANE | 14(1.1) | 4.4(0.7) | 6.4 | 2.1 | |
| 3-METHYLHEPTANE | 6.5(0.5) | 5.1(0.7) | 8.2 | 3.4(0.7) | 2.2(1.3) |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 |
| OCTANE | 14(1.1) | 9.8(1.0) | 10 | 0 | 0 |
| ETHYLCYCLOHEXANE | 4.9(1.1) | 4.5(0.5) | 5.6 | 3.2(0.6) | 2.3(1.0) |
| ETHYLBENZENE | 2.2(0.2) | 1.1(0.1) | 1.5 | 1.0(0.2) | 1.2 |
| M-XYLENE | 3.9(0.8) | 1.0(0.3) | 1.0 | 0 | 0 |
| P-XYLENE | 3.0(0.1) | 2.4(0.3) | 3.0 | 2.1(0.3) | 1.5(0.6) |
| O-XYLENE | 1.9(0.3) | 0 | 0 | 0 | 0 |
| NONANE | 21(2.0) | 18(2.5) | 18 | 4.1(1.1) | 1.9(0.7) |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 7.5(0.2) | 6.7(0.7) | 7.4 | 5.3(0.7) | 3.8(2.8) |
| 1,2,4-TRIMETHYLBENZENE | 13(1.0) | 12(0.7) | 17 | 10(7.5) | 7.0(3.5) |

TABLE B-3. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER STERILE BOTTLE TESTS (CONCLUDED).

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| DECANE | 30(2.7) | 27(3.7) | 30 | 13(3.5) | 7.5(3.5) |
| 1,2,3-TRIMETHYLBENZENE | 4.3(0.7) | 1.4(0.2) | 4.7 | 4.1(0.5) | 4.9 |
| INDAN | 1.9(0.1) | 1.5(0.2) | 1.9 | 1.5(0.2) | 1.4(0.4) |
| UNDECANE | 45(4.9) | 43(5.5) | 53 | 33(8.0) | 23(9.9) |
| NAPHTHALENE | 11(1.1) | 9.7(1.0) | 12 | 8.4(1.3) | 7.1(3.3) |
| DODECANE | 42(4.3) | 49(5.6) | 64 | 50(7.9) | 44(16) |
| TRIDECANE | 39(2.8) | 42(5.9) | 51 | 46(8.7) | 48(17) |
| TETRADECANE | 20(1.9) | 22(3.2) | 30 | 28(3.2) | 30(10) |
| PENTADECANE | 6.5(0.8) | 7.1(1.1) | 9.5 | 8.6(1.2) | 9.8(2.7) |
| HEXADECANE | 0.5(0.6) | 0.3(.08) | 0.7 | 0.5(0.3) | 0.7(0.2) |

[a] Values are means (± the standard deviations). Where no standard deviation is indicated, n=1; i.e., hydrocarbon concentrations in the other samples was below detection limits. Only one bottle was analyzed for Day 8 samples.

[b] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-4. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER STERILE BOTTLE TESTS.

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| BENZENE | 0 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 1.4 | 0 | 0 | 0 | 0 |
| HEPTANE | 27(7.5) | 26(2.5) | 19 | 19(6.9) | 11 |
| METHYLCYCLOHEXANE | 1.9(0.8) | 1.3 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE | 3.7(2.1) | 2.5(0.9) | 7.8 | 2.9(0.1) | 2.6(0.3) |
| 2-METHYLHEPTANE | 5.0(2.4) | 5.5(3.4) | 4.6 | 3.7(0.6) | 3.5(2.4) |
| 3-METHYLHEPTANE | 6.4(3.2) | 4.3(2.5) | 5.9 | 4.5(0.7) | 4.3(2.7) |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 |
| OCTANE | 14(6.9) | 12(7.3) | 13.8 | 10(1.7) | 10(6.6) |
| ETHYLCYCLOHEXANE | 4.9(2.3) | 4.1(2.4) | 4.3 | 3.1(0.5) | 2.9(1.8) |
| ETHYLBENZENE | 1.8(0.8) | 1.3(0.1) | 0 | .9 | 1.2 |
| M-XYLENE | 2.7(1.5) | 1.0(0.1) | 0 | 0 | 0 |
| P-XYLENE | 2.8(1.4) | 2.2(0.9) | 2.2 | 1.9(0.3) | 1.9(0.1) |
| O-XYLENE | 3.5(1.3) | 0 | 0 | 0 | 0 |
| NONANE | 24(9.5) | 20(9.8) | 23 | 15.9(10) | 21(10) |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 6.0(9.6) | 5.3(2.5) | 5.4 | 4.7(0.6) | 4.7(2.2) |
| 1,2,4-TRIMETHYLBENZENE | 15(5.5) | 12(5.4) | 13 | 8.8(6.1) | 12(5.9) |

TABLE B-4. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER STERILE BOTTLE TESTS (CONCLUDED).

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| DECANE | 39(10) | 31(10) | 35 | 33(3.6) | 34(11) |
| 1,2,3-TRIMETHYLBENZENE | 3.5(1.9) | 4.0 | 0 | 0 | 0 |
| INDAN | 2.2(0.8) | 1.6(0.1) | 1.4 | 1.4(0.2) | 1.4(0.5) |
| UNDECANE | 68(14) | 55(7.4) | 60 | 56(5.9) | 58(13) |
| NAPHTHALENE | 16(5.2) | 11(3.4) | 9.2 | 9.1(1.3) | 10(2.9) |
| DODECANE | 78(11) | 64(11) | 68 | 65(7.0) | 69(14) |
| TRIDECANE | 67(7.5) | 59(8.4) | 57 | 59(4.5) | 61(11) |
| TETRADECANE | 36(4.2) | 31(4.5) | 31 | 32(4.0) | 34(5.8) |
| PENTADECANE | 11(1.3) | 9.6(1.4) | 9.7 | 9.4(1.2) | 10(1.4) |
| HEXADECANE | 0.9(0.1) | 1.6(1.7) | 0.7 | 0.5(0.8) | 0.8(0.2) |

[a] Values are means (± the standard deviations). Where no standard deviation is indicated, n=1; i.e., hydrocarbon concentrations in the other samples was below detection limits. Only one bottle was analyzed for Day 8 samples.

[b] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-5. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER TRAYS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 7 | 14 | 21 | 29 |
| BENZENE | 0 | 0 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 5.4 | 0 | 4.9 | 1.5 | 0 | 1.7 |
| 2,3-DIMETHYLPENTANE | 2.9 | 0 | 3.3 | 1.3 | 0 | 1.2 |
| 3-METHYLHEXANE | 11.1 | 1.9 | 12.6 | 5.0 | 0 | 4.7 |
| HEPTANE | 114 | 18.4 | 124 | 47.0 | 0 | 47.5 |
| METHYLCYCLOHEXANE | 16.5 | 2.4 | 18.1 | 6.7 | 0 | 6.6 |
| 2,5-DIMETHYLHEXANE | 4.1 | 0 | 4.9 | 2.1 | 0 | 1.8 |
| 2,4-DIMETHYLHEXANE | 6.4 | 0 | 7.6 | 3.2 | 0 | 2.9 |
| METHYLBENZENE | 6.1 | 0 | 3.0 | 1.7 | 0 | 0 |
| 2-METHYLHEPTANE | 31.6 | 5.2 | 37.3 | 15.1 | 1.2 | 14.7 |
| 3-METHYLHEPTANE | 36.1 | 6.8 | 43.4 | 17.8 | 1.7 | 17.0 |
| 1,1-DIMETHYLCYCLOHEXANE | 1.5 | 0 | 1.9 | 0 | 0 | 0 |
| OCTANE | 68.0 | 11.6 | 81.6 | 32.2 | 2.6 | 31.9 |
| ETHYLCYCLOHEXANE | 21.5 | 3.9 | 27.2 | 10.9 | 1.1 | 10.8 |
| ETHYLBENZENE | 7.6 | 1.1 | 7.6 | 3.2 | 0 | 2.5 |
| M-XYLENE | 13.7 | 1.4 | 11.1 | 4.7 | 0 | 2.6 |
| P-XYLENE | 12.3 | 1.9 | 14.0 | 5.5 | 0 | 4.9 |
| O-XYLENE | 10.4 | 0 | 10.0 | 3.0 | 0 | 2.3 |
| NONANE | 69.2 | 13.1 | 95.0 | 36.9 | 3.6 | 37.3 |
| ISOPROPYLBENZENE (CUMENE) | 1.9 | 0 | 2.0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 23.8 | 4.2 | 33.1 | 11.8 | 2.2 | 12.4 |
| 1,2,4-TRIMETHYLBENZENE | 32.3 | 4.9 | 38.7 | 10.8 | 0 | 13.5 |

TABLE B-5. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER TRAYS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 7 | 14 | 21 | 29 |
| DECANE | 84.4 | 17.3 | 126 | 48.6 | 4.9 | 47.3 |
| 1,2,3-TRIMETHYLBENZENE | 14.5 | 2.1 | 17.7 | 4.3 | 0 | 5.5 |
| INDAN | 6.1 | 1.0 | 8.0 | 2.5 | 0 | 2.7 |
| UNDECANE | 127 | 27.7 | 197 | 74.3 | 7.7 | 77.5 |
| NAPHTHALENE | 37.6 | 6.0 | 50.8 | 15.3 | 1.8 | 16.3 |
| DODECANE | 155 | 36.3 | 256 | 93.2 | 10.4 | 89.8 |
| TRIDECANE | 145 | 32.7 | 238 | 85.7 | 9.5 | 82.7 |
| TETRADECANE | 89.0 | 20.5 | 152 | 52.7 | 6.1 | 50.8 |
| PENTADECANE | 26.4 | 6.8 | 48.8 | 16.5 | 2.3 | 16.9 |
| HEXADECANE | 3.7 | 0 | 1.0 | 0.9 | 0 | 1.3 |

[a] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-6. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER TRAYS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 10 | 14 | 21 | 37 |
| BENZENE | 0 | 0 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 1.7 | 12.2 | 3.3 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 6.5 | 1.9 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 3.8 | 25.3 | 7.4 | 1.4 | 1.4 | 1.0 |
| HEPTANE | 37.9 | 254 | 78.1 | 14.3 | 11.3 | 10.2 |
| METHYLCYCLOHEXANE | 5.6 | 36.5 | 10.9 | 1.2 | 0 | 1.4 |
| 2,5-DIMETHYLHEXANE | 1.4 | 9.2 | 2.8 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 2.2 | 14.0 | 4.3 | 0 | 1.3 | 0 |
| METHYLBENZENE | 2.1 | 12.6 | 2.0 | 0 | 0.9 | 1.7 |
| 2-METHYLHEPTANE | 10.6 | 68.8 | 21.6 | 4.0 | 4.8 | 2.9 |
| 3-METHYLHEPTANE | 13.3 | 78.5 | 24.6 | 5.0 | 6.1 | 3.6 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 3.3 | 0 | 0 | 0 | 0 |
| OCTANE | 24.6 | 147 | 46.9 | 8.3 | 9.6 | 6.6 |
| ETHYLCYCLOHEXANE | 8.0 | 45.8 | 14.5 | 2.6 | 3.6 | 2.2 |
| ETHYLBENZENE | 2.9 | 16.2 | 4.5 | 0 | 1.1 | 0 |
| M-XYLENE | 5.2 | 29.0 | 7.6 | 0 | 1.1 | 0.9 |
| P-XYLENE | 4.7 | 26.4 | 7.7 | 1.1 | 1.8 | 1.0 |
| O-XYLENE | 2.9 | 24.3 | 5.8 | 0 | 0 | 0 |
| NONANE | 26.8 | 149 | 46.8 | 7.7 | 11.4 | 7.1 |
| ISOPROPYLBENZENE (CUMENE) | 0 | 4.9 | 1.2 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 9.5 | 51.4 | 15.8 | 1.7 | 2.7 | 2.3 |
| 1,2,4-TRIMETHYLBENZENE | 11.9 | 74.6 | 22.3 | 0 | 0.9 | 14.5 |

TABLE B-6. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER TRAYS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 10 | 14 | 21 | 37 |
| DECANE | 34.6 | 188 | 56.8 | 8.6 | 13.5 | 9.4 |
| 1,2,3-TRIMETHYLBENZENE | 5.6 | 34.8 | 9.3 | 0 | 0 | 4.9 |
| INDAN | 2.4 | 16.3 | 4.0 | 0 | 0 | 0 |
| UNDECANE | 54.7 | 283 | 84.9 | 11.8 | 18.7 | 14.6 |
| NAPHTHALENE | 13.0 | 85.8 | 26.4 | 1.9 | 3.2 | 3.2 |
| DODECANE | 66.1 | 406 | 107 | 13.8 | 22.2 | 20.2 |
| TRIDECANE | 59.1 | 366 | 98.5 | 11.2 | 17.8 | 19.2 |
| TETRADECANE | 35.1 | 221 | 56.6 | 6.7 | 11.0 | 11.8 |
| PENTADECANE | 10.5 | 68.4 | 17.4 | 2.5 | 4.1 | 3.9 |
| HEXADECANE | 1.4 | 9.1 | 1.9 | 0 | 0 | 0 |

[a] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-7. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER SITE IN THE FIELD.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 7 | 14 | 21 | 28 | 36 |
| BENZENE | 1.8 | 0 | 0 | 0 | 0 | 0 |
| CYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 1.9 | 1.6 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 7.1 | 6.8 | 0 | 0 | 0 | 0 |
| HEPTANE | 72.8 | 69.5 | 0 | 6.3 | 7.2 | 0 |
| METHYLCYCLOHEXANE | 8.4 | 4.8 | 0 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 2.5 | 2.9 | 0 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 3.9 | 4.6 | 0 | 0 | 0 | 0 |
| METHYLBENZENE | 0 | 0 | 0 | 0 | 0 | 1.8 |
| 2-METHYLHEPTANE | 17.9 | 20.3 | 1.7 | 2.1 | 2.4 | 0 |
| 3-METHYLHEPTANE | 20.3 | 23.0 | 2.2 | 2.8 | 3.0 | 0 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| OCTANE | 36.2 | 39.2 | 3.6 | 4.5 | 4.3 | 0 |
| ETHYLCYCLOHEXANE | 10.4 | 10.9 | 1.3 | 1.7 | 1.6 | 1.0 |
| ETHYLBENZENE | 2.0 | 1.9 | 0 | 3.6 | 0 | 0 |
| M-XYLENE | 1.3 | 0 | 0 | 0 | 0 | 0.9 |
| P-XYLENE | 4.0 | 4.6 | 0 | 0.9 | 0 | 0 |
| O-XYLENE | 1.3 | 0 | 0 | 0 | 0 | 0 |
| NONANE | 29.3 | 32.9 | 8.4 | 5.9 | 4.1 | 2.1 |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 3.6 | 6.4 | 1.2 | 1.5 | 1.0 | 1.0 |
| 1,2,4-TRIMETHYLBENZENE | 5.2 | 0.9 | 0 | 0 | 0 | 0 |

TABLE B-7. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE SHALLOW WATER SITE IN THE FIELD (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 7 | 14 | 21 | 28 | 36 |
| DECANE | 28.3 | 31.9 | 6.8 | 8.0 | 4.6 | 3.2 |
| 1,2,3-TRIMETHYLBENZENE | 1.9 | 0 | 0 | 0 | 0 | 0 |
| INDAN | 1.3 | 1.3 | 0 | 0 | 0 | 0 |
| UNDECANE | 36.9 | 41.6 | 10.5 | 12.5 | 8.2 | 5.1 |
| NAPHTHALENE | 6.7 | 6.0 | 1.6 | 2.2 | 1.7 | 1.5 |
| DODECANE | 37.5 | 43.7 | 13.1 | 16.9 | 10.3 | 6.4 |
| TRIDECANE | 28.7 | 35.1 | 11.3 | 15.4 | 7.8 | 4.6 |
| TETRADECANE | 16.8 | 20.9 | 6.8 | 9.9 | 5.0 | 2.6 |
| PENTADECANE | 4.8 | 6.5 | 2.6 | 3.7 | 1.7 | 0.9 |
| HEXADECANE | 2.1 | 0 | 0 | 0 | 0 | 0 |

[a] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-8. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER SITE IN THE FIELD.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 4 | 7 | 10 | 14 | 21 |
| BENZENE | 0 | 0 | 0 | 0 | 1.2 | 0 |
| CYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | 1.5 | 0 |
| 3-METHYLHEXANE | 2.9 | 3.1 | 2.8 | 0 | 5.9 | 1.1 |
| HEPTANE | 29.7 | 30.0 | 28.8 | 6.8 | 56.9 | 11.3 |
| METHYLCYCLOHEXANE | 4.2 | 4.1 | 3.4 | 0 | 6.7 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 2.7 | 0 |
| 2,4-DIMETHYLHEXANE | 1.4 | 1.5 | 1.4 | 0 | 4.2 | 0 |
| METHYLBENZENE | 1.2 | 1.1 | 0 | 0 | 2.2 | 0 |
| 2-METHYLHEPTANE | 6.8 | 6.3 | 6.8 | 1.7 | 18.3 | 3.8 |
| 3-METHYLHEPTANE | 8.1 | 7.6 | 8.4 | 2.0 | 21.0 | 4.8 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| OCTANE | 14.4 | 13.1 | 15.1 | 3.5 | 36.2 | 7.5 |
| ETHYLCYCLOHEXANE | 4.3 | 3.7 | 4.5 | 1.0 | 11.3 | 2.5 |
| ETHYLBENZENE | 1.4 | 1.3 | 1.4 | 0 | 2.8 | 0 |
| M-XYLENE | 2.8 | 2.2 | 2.3 | 0 | 3.1 | 0 |
| P-XYLENE | 2.2 | 1.9 | 2.3 | 0 | 5.0 | 1.2 |
| O-XYLENE | 0 | 1.1 | 1.1 | 0 | 1.8 | 0 |
| NONANE | 12.2 | 9.3 | 13.7 | 3.2 | 32.9 | 8.0 |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 3.4 | 2.6 | 4.3 | 0 | 3.1 | 1.6 |
| 1,2,4-TRIMETHYLBENZENE | 4.3 | 3.3 | 4.9 | 0 | 3.7 | 0 |

TABLE B-8. ACTUAL HYDROCARBON CONCENTRATIONS (mg/ℓ OF EXTRACT)[a] IN SAMPLES TAKEN FROM THE DEEP WATER SITE IN THE FIELD (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 4 | 7 | 10 | 14 | 21 |
| DECANE | 12.7 | 8.3 | 15.7 | 3.1 | 35.0 | 8.6 |
| 1,2,3-TRIMETHYLBENZENE | 1.5 | 1.3 | 2.5 | 0 | 1.4 | 0 |
| INDAN | 0 | 0 | 1.1 | 0 | 1.5 | 0 |
| UNDECANE | 16.0 | 9.5 | 23.3 | 3.9 | 46.3 | 11.1 |
| NAPHTHALENE | 3.8 | 2.0 | 7.5 | 0 | 7.4 | 1.7 |
| DODECANE | 18.4 | 9.9 | 31.4 | 4.3 | 49.1 | 12.4 |
| TRIDECANE | 14.5 | 7.7 | 27.4 | 3.3 | 40.8 | 10.7 |
| TETRADECANE | 9.1 | 4.6 | 18.6 | 1.9 | 23.3 | 6.0 |
| PENTADECANE | 2.8 | 1.6 | 6.4 | 0 | 7.1 | 2.4 |
| HEXADECANE | 0 | 0 | 0 | 0 | 0.4 | 0 |

[a] Zero reflects a hydrocarbon concentration below detection limit, either because of sample size or actual disappearance.

TABLE B-9. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER ACTIVE BOTTLES TESTS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| BENZENE | NS[a] | NS | NS | NS | NS | NS |
| CYCLOHEXANE | 0[b] | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | NS | NS | NS |
| 3-METHYLHEXANE | 7 | 4 | 9 | 3 | 3 | 0 |
| HEPTANE | 1 | 0 | 1 | 0 | 0 | 0 |
| METHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | NS | NS | NS |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 24 | 15 | 4 | 16 | NS | 6 |
| 2-METHYLHEPTANE | 3 | 2 | 5 | 0 | 0 | 0 |
| 3-METHYLHEPTANE | 4 | 3 | 6 | 2 | 2 | 0 |
| 1,1-DIMETHYLCYCLOHEXANE | NS | NS | NS | NS | NS | NS |
| OCTANE | 6 | 3 | 4 | 0 | 0 | 0 |
| ETHYLCYCLOHEXANE | 8 | 5 | 10 | 4 | 4 | 0 |
| ETHYLBENZENE | 0 | 0 | 0 | 0 | 0 | 0 |
| M-XYLENE | 3 | 0 | 0 | 0 | 0 | 0 |
| P-XYLENE | 7 | 4 | 8 | 0 | NS | 0 |
| O-XYLENE | 0 | 0 | 0 | 0 | 0 | 0 |
| NONANE | 20 | 12 | 13 | 0 | 3 | 0 |
| ISOPROPYLBENZENE (CUMENE) | NS | NS | NS | NS | NS | NS |
| 1,3,5-TRIMETHYLBENZENE | 21 | 12 | 20 | 11 | 15 | 0 |
| 1,2.4-TRIMETHYLBENZENE | 5 | 3 | 5 | 4 | 6 | 0 |

TABLE B-9. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER ACTIVE BOTTLES TESTS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| DECANE | 43 | 28 | 34 | 10 | 11 | 0 |
| 1,2,3-TRIMETHYLBENZENE | 11 | 0 | 0 | NS | NS | NS |
| INDAN | 26 | 30 | 23 | 28 | 36 | NS |
| UNDECANE | 64 | 51 | 61 | 28 | 25 | 9 |
| NAPHTHALENE | 58 | 37 | 38 | 28 | 30 | 17 |
| DODECANE | 84 | 78 | 83 | 59 | 61 | 40 |
| TRIDECANE | 95 | 88 | 94 | 78 | 89 | 70 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 108 | 98 | 95 | 107 | 101 | 118 |
| HEXADECANE | 143 | 93 | 86 | 131 | NS | NS |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-10. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER STERILE BOTTLES TESTS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| BENZENE | 31 | 0 | NS[a] | NS | NS | NS |
| CYCLOHEXANE | 0[b] | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | NS | NS |
| 3-METHYLHEXANE | 4 | 5 | 4 | 3 | 0 | 3 |
| HEPTANE | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYLCYCLOHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | NS | NS | NS |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 0 | 14 | 7 | 19 | NS | 8 |
| 2-METHYLHEPTANE | 2 | 3 | 2 | 0 | 0 | 1 |
| 3-METHYLHEPTANE | 2 | 3 | 2 | 2 | 0 | 2 |
| 1,1-DIMETHYLCYCLOHEXANE | NS | NS | NS | NS | NS | NS |
| OCTANE | 3 | 5 | 3 | 2 | 2 | 3 |
| ETHYLCYCLOHEXANE | 5 | 6 | 4 | 0 | 0 | NS |
| ETHYLBENZENE | 0 | 0 | 0 | 0 | 0 | ns |
| M-XYLENE | 3 | 0 | 0 | 0 | 0 | 0 |
| P-XYLENE | 4 | 6 | 4 | 0 | NS | NS |
| O-XYLENE | 0 | 0 | 0 | 0 | 0 | 0 |
| NONANE | 13 | 17 | 13 | 10 | 12 | 17 |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | 0 | 0 |
| 1,3,5-TRIMETHYLBENZENE | 14 | 15 | 11 | 8 | 9 | 13 |
| 1,2,4-TRIMETHYLBENZENE | 3 | 4 | 3 | 3 | 3 | 5 |

TABLE B-10. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER STERILE BOTTLES TESTS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 4 | 8 | 17 | 29 |
| DECANE | 33 | 36 | 33 | 26 | 30 | 40 |
| 1,2,3-TRIMETHYLBENZENE | 7 | 0 | 0 | 0 | NS | NS |
| INDAN | 19 | 18 | 16 | 0 | 26 | 23 |
| UNDECANE | 55 | 57 | 56 | 46 | 53 | 64 |
| NAPHTHALENE | 43 | 39 | 35 | 26 | 30 | 34 |
| DODECANE | 77 | 80 | 79 | 68 | 75 | 81 |
| TRIDECANE | 92 | 93 | 93 | 85 | 89 | 88 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 108 | 100 | 99 | 107 | 101 | 99 |
| HEXADECANE | 125 | 100 | 111 | 125 | NS | 106 |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-11. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER ACTIVE BOTTLES TESTS.

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| BENZENE | 22 | 0 | NS[a] | NS | NS |
| CYCLOHEXANE | 0[b] | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | NS |
| 3-METHYLHEXANE | 12 | 12 | 10 | 5 | 0 |
| HEPTANE | 2 | 2 | 0 | 0 | 0 |
| METHYLCYCLOHEXANE | 1 | 1 | 1 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | NS | NS |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 2 | 2 | 1 | 0 | 6 |
| 2-METHYLHEPTANE | 6 | 7 | 5 | 0 | 0 |
| 3-METHYLHEPTANE | 8 | 8 | 6 | 3 | 0 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | NS | NS | NS | NS |
| OCTANE | 8 | 6 | 1 | 0 | 0 |
| ETHYLCYCLOHEXANE | 13 | 13 | 11 | 7 | 4 |
| ETHYLBENZENE | 8 | 7 | 12 | 5 | 0 |
| M-XYLENE | 5 | 2 | 0 | 0 | 0 |
| P-XYLENE | 13 | 12 | 10 | 8 | 6 |
| O-XYLENE | 0 | 0 | 0 | 0 | 0 |
| NONANE | 27 | 20 | 6 | 4 | 0 |
| ISOPROPYLBENZENE (CUMENE) | 0 | NS | NS | NS | NS |
| 1,3,5-TRIMETHYLBENZENE | 29 | 26 | 25 | 20 | 12 |
| 1,2,4-TRIMETHYLBENZENE | 10 | 4 | 6 | 5 | 4 |

TABLE B-11. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER ACTIVE BOTTLES TESTS (CONCLUDED).

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| DECANE | 52 | 41 | 24 | 16 | 6 |
| 1,2,3-TRIMETHYLBENZENE | 9 | 5 | 0 | 0 | 0 |
| INDAN | 32 | 30 | 25 | 27 | 24 |
| UNDECANE | 74 | 65 | 49 | 38 | 23 |
| NAPHTHALENE | 51 | 50 | 44 | 34 | 26 |
| DODECANE | 87 | 84 | 76 | 69 | 55 |
| TRIDECANE | 97 | 94 | 90 | 83 | 84 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 102 | 98 | 100 | 100 | 109 |
| HEXADECANE | NS | 97 | 93 | 81 | 106 |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-12. RATIOS OF (NORMALIZED AS PERCENT OF STANDARD) CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER STERILE BOTTLE TESTS.

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| BENZENE | NS[a] | NS | NS | NS | NS |
| CYCLOHEXANE | 0[b] | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 4 | 11 | 8 | 6 | 6 |
| HEPTANE | 0 | 4 | 3 | 4 | 0 |
| METHYLCYCLOHEXANE | 6 | 1 | 10 | 0 | 4 |
| 2,5-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 0 | 0 | 0 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 8 | 2 | 14 | 0 | 5 |
| 2-METHYLHEPTANE | 2 | 6 | 4 | 3 | 3 |
| 3-METHYLHEPTANE | 3 | 7 | 5 | 3 | 3 |
| 1,1-DIMETHYLCYCLOHEXANE | NS | NS | NS | NS | NS |
| OCTANE | 4 | 10 | 7 | 5 | 5 |
| ETHYLCYCLOHEXANE | 5 | 12 | 8 | 6 | 6 |
| ETHYLBENZENE | 5 | 6 | 0 | 0 | 0 |
| M-XYLENE | 4 | 2 | 0 | 0 | 0 |
| P-XYLENE | 5 | 10 | 7 | 7 | 7 |
| O-XYLENE | 0 | 0 | 0 | 157 | 0 |
| NONANE | 15 | 27 | 21 | 20 | 21 |
| ISOPROPYLBENZENE (CUMENE) | 0 | NS | NS | NS | NS |
| 1,3,5-TRIMETHYLBENZENE | 8 | 23 | 16 | 16 | 16 |
| 1,2,4-TRIMETHYLBENZENE | 10 | 7 | 4 | 4 | 4 |

TABLE B-12. RATIOS OF (NORMALIZED AS PERCENT OF STANDARD) CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER STERILE BOTTLE TESTS (CONCLUDED).

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 2 | 4 | 8 | 17 | 29 |
| DECANE | 38 | 49 | 40 | 42 | 41 |
| 1,2,3-TRIMETHYLBENZENE | 9 | 5 | 0 | 0 | 0 |
| INDAN | 22 | 26 | 19 | 22 | 21 |
| UNDECANE | 66 | 71 | 61 | 67 | 64 |
| NAPHTHALENE | 55 | 50 | 33 | 35 | 34 |
| DODECANE | 86 | 86 | 77 | 82 | 80 |
| TRIDECANE | 94 | 95 | 91 | 93 | 93 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 98 | 100 | 103 | 93 | 98 |
| HEXADECANE | 102 | 104 | 111 | 79 | 106 |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-13. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER TRAYS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 7 | 14 | 21 | 29 |
| BENZENE | NS[a] | NS | 0[b] | NS | NS | NS |
| CYCLOHEXANE | 6 | 0 | 2 | 2 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 8 | 0 | 3 | 4 | 0 | 0 |
| 3-METHYLHEXANE | 9 | 4 | 4 | 4 | 12 | 10 |
| HEPTANE | 10 | 5 | 4 | 5 | NS | 3 |
| METHYLCYCLOHEXANE | 10 | 4 | 4 | 5 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 14 | 0 | 7 | 8 | 0 | NS |
| 2,4-DIMETHYLHEXANE | 13 | 0 | 6 | 7 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 14 | 6 | 6 | 6 | 0 | 0 |
| 2-METHYLHEPTANE | 16 | 8 | 8 | 9 | 6 | 5 |
| 3-METHYLHEPTANE | 5 | 2 | 2 | 2 | 8 | 6 |
| 1,1-DIMETHYLCYCLOHEXANE | NS | NS | NS | 0 | NS | NS |
| OCTANE | 19 | 11 | 10 | 11 | 7 | 7 |
| ETHYLCYCLOHEXANE | 21 | 13 | 12 | 14 | 12 | 10 |
| ETHYLBENZENE | 18 | 8 | 8 | 10 | 0 | 0 |
| M-XYLENE | 17 | 6 | 6 | 8 | 0 | 0 |
| P-XYLENE | 21 | 11 | 11 | 12 | NS | 8 |
| O-XYLENE | 19 | 0 | 8 | 4 | 0 | 0 |
| NONANE | 32 | 21 | 21 | 23 | 19 | 15 |
| ISOPROPYLBENZENE (CUMENE) | 25 | NS | 13 | 0 | NS | NS |
| 1,3,5-TRIMETHYLBENZENE | 36 | 23 | 16 | 14 | 41 | 18 |
| 1,2,4-TRIMETHYLBENZENE | 36 | 19 | 20 | 16 | 0 | 8 |

TABLE B-13. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER TRAYS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 7 | 14 | 21 | 29 |
| DECANE | 47 | 36 | 36 | 39 | 34 | 29 |
| 1,2,3-TRIMETHYLBENZENE | 29 | 16 | 19 | 13 | 0 | 14 |
| INDAN | 42 | 26 | 32 | 35 | 25 | 0 |
| UNDECANE | 62 | 53 | 50 | 55 | 49 | 45 |
| NAPHTHALENE | 60 | 47 | 37 | 41 | 28 | 31 |
| DODECANE | 76 | 73 | 68 | 70 | 70 | 67 |
| TRIDECANE | 93 | 86 | 89 | 88 | 84 | 81 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 101 | 106 | 97 | 101 | 122 | 121 |
| HEXADECANE | 111 | 0 | 98 | 93 | NS | 140 |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-14. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER TRAYS.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 10 | 14 | 21 | 37 |
| BENZENE | 0[b] | 0 | 0 | 0 | 0 | NS[a] |
| CYCLOHEXANE | 3 | 4 | 4 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 0 | 6 | 6 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 6 | 6 | 6 | 10 | 6 | 4 |
| HEPTANE | 7 | 7 | 8 | 12 | 5 | 5 |
| METHYLCYCLOHEXANE | 7 | 7 | 8 | 6 | 4 | 4 |
| 2,5-DIMETHYLHEXANE | 10 | 10 | 11 | 0 | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 9 | 10 | 10 | NS | 15 | 0 |
| METHYLBENZENE (TOLUENE) | 9 | 10 | 11 | 9 | 5 | 6 |
| 2-METHYLHEPTANE | 10 | 12 | 13 | 20 | 14 | 8 |
| 3-METHYLHEPTANE | 3 | 4 | 4 | 6 | 4 | 2 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | NS | NS | NS | NS | NS |
| OCTANE | 14 | 14 | 16 | 23 | 16 | 8 |
| ETHYLCYCLOHEXANE | 17 | 16 | 18 | 26 | 22 | 11 |
| ETHYLBENZENE | 14 | 13 | 13 | NS | 16 | 13 |
| M-XYLENE | 13 | 13 | 12 | 0 | 9 | 6 |
| P-XYLENE | 16 | 16 | 17 | 20 | 20 | 11 |
| O-XYLENE | 11 | 16 | 14 | 0 | 0 | 0 |
| NONANE | 26 | 25 | 28 | 38 | 35 | 20 |
| ISOPROPYLBENZENE (CUMENE) | NS | 23 | 22 | 0 | NS | NS |
| 1,3,5-TRIMETHYLBENZENE | 31 | 29 | 33 | 29 | 29 | 22 |
| 1,2,4-TRIMETHYLBENZENE | 28 | 29 | 34 | NS | 7 | 16 |

TABLE B-14. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER TRAYS (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 3 | 4 | 10 | 14 | 21 | 37 |
| DECANE | 41 | 39 | 44 | 55 | 52 | 34 |
| 1,2,3-TRIMETHYLBENZENE | 24 | 26 | 26 | NS | NS | 16 |
| INDAN | 34 | 40 | 36 | NS | NS | NS |
| UNDECANE | 57 | 52 | 60 | 69 | 66 | 98 |
| NAPHTHALENE | 46 | 45 | 55 | 37 | 35 | 34 |
| DODECANE | 75 | 66 | 75 | 85 | 82 | 70 |
| TRIDECANE | 90 | 88 | 91 | 91 | 92 | 90 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 104 | 98 | 109 | 123 | 113 | 107 |
| HEXADECANE | 109 | 0 | 106 | NS | NS | NS |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-15. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER SITE IN THE FIELD (CONTINUED).

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 1 | 7 | 20 | 27 | 35 |
| BENZENE | 18 | 0 | NS[a] | NS | NS |
| CYCLOHEXANE | 0[b] | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 20 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 21 | 26 | 0 | 0 | 0 |
| HEPTANE | 26 | 31 | 0 | 0 | 0 |
| METHYLCYCLOHEXANE | 20 | 25 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | 34 | 0 | NS | NS | 0 |
| 2,4-DIMETHYLHEXANE | 31 | 32 | 0 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 28 | 34 | 0 | 0 | 0 |
| 2-METHYLHEPTANE | 36 | 36 | 0 | 0 | 0 |
| 3-METHYLHEPTANE | 13 | 17 | 0 | 0 | 0 |
| 1,1-DIMETHYLCYCLOHEXANE | 0 | NS | NS | NS | NS |
| OCTANE | 41 | 43 | 0 | 0 | 0 |
| ETHYLCYCLOHEXANE | 43 | 45 | 0 | 0 | NS |
| ETHYLBENZENE | 19 | 0 | 0 | 0 | 0 |
| M-XYLENE | 6 | 14 | NS | 0 | NS |
| P-XYLENE | 29 | 30 | 0 | 0 | 0 |
| O-XYLENE | 10 | 0 | 0 | 0 | 0 |
| NONANE | 56 | 54 | 16 | 26 | 32 |
| ISOPROPYLBENZENE (CUMENE) | 0 | 0 | 0 | 0 | NS |
| 1,3,5-TRIMETHYLBENZENE | 23 | 20 | 0 | 0 | NS |
| 1,2,4-TRIMETHYLBENZENE | 24 | 28 | 0 | 0 | 0 |

TABLE B-15. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE SHALLOW WATER SITE IN THE FIELD (CONCLUDED).

| | DAYS | | | | |
|---|---|---|---|---|---|
| | 1 | 7 | 20 | 27 | 35 |
| DECANE | 67 | 65 | 33 | 47 | 51 |
| 1,2,3-TRIMETHYLBENZENE | 16 | 27 | 0 | 0 | 0 |
| INDAN | 38 | 0 | 0 | 0 | NS |
| UNDECANE | 77 | 72 | 49 | 67 | 74 |
| NAPHTHALENE | 46 | 47 | 0 | 49 | 52 |
| DODECANE | 85 | 86 | 70 | 83 | 78 |
| TRIDECANE | 87 | 90 | 88 | 96 | 92 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 100 | 118 | 111 | 0 | 112 |
| HEXADECANE | 153 | NS | NS | NS | NS |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.

TABLE B-16. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER SITE IN THE FIELD.

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 4 | 7 | 10 | 14 | 20 |
| BENZENE | NS[a] | NS | NS | NS | NS | 0[b] |
| CYCLOHEXANE | 22 | 0 | 0 | 0 | 0 | 0 |
| 2,3-DIMETHYLPENTANE | 31 | 0 | 0 | 0 | 0 | 0 |
| 3-METHYLHEXANE | 32 | 33 | 57 | 48 | 17 | 10 |
| HEPTANE | 36 | 38 | 22 | 20 | 20 | 11 |
| METHYLCYCLOHEXANE | 34 | 0 | 0 | 0 | 0 | 0 |
| 2,5-DIMETHYLHEXANE | NS | NS | NS | NS | 0 | 0 |
| 2,4-DIMETHYLHEXANE | 40 | 42 | 0 | 0 | 0 | 0 |
| METHYLBENZENE (TOLUENE) | 47 | 50 | 0 | 0 | 0 | 0 |
| 2-METHYLHEPTANE | 43 | 46 | 29 | 29 | 32 | 22 |
| 3-METHYLHEPTANE | 20 | 21 | 36 | 31 | 11 | 6 |
| 1,1-DIMETHYLCYCLOHEXANE | NS | NS | NS | NS | NS | 0 |
| OCTANE | 50 | 54 | 38 | 34 | 38 | 25 |
| ETHYLCYCLOHEXANE | 51 | 57 | 40 | 36 | 42 | 29 |
| ETHYLBENZENE | 39 | 45 | 0 | 0 | 0 | 0 |
| M-XYLENE | 38 | 42 | 0 | 0 | 0 | 11 |
| P-XYLENE | 45 | 50 | 0 | 0 | 33 | 26 |
| O-XYLENE | 24 | 30 | 0 | 0 | 0 | 0 |
| NONANE | 61 | 66 | 57 | 54 | 59 | 47 |
| ISOPROPYLBENZENE (CUMENE) | NS | NS | NS | NS | NS | 0 |
| 1,3,5-TRIMETHYLBENZENE | 55 | 62 | 38 | NS | 42 | 32 |
| 1,2,4-TRIMETHYLBENZENE | 49 | 57 | 0 | 0 | 0 | 0 |

TABLE B-16. RATIOS (NORMALIZED AS PERCENT OF STANDARD) OF CONCENTRATIONS OF SELECTED HYDROCARBONS TO CONCENTRATIONS OF TETRADECANE IN SAMPLES TAKEN FROM THE DEEP WATER SITE IN THE FIELD (CONCLUDED).

| | DAYS | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 4 | 7 | 10 | 14 | 20 |
| DECANE | 68 | 74 | 77 | 68 | 76 | 64 |
| 1/2/3-TRIMETHYLBENZENE | 44 | 45 | 0 | 0 | 0 | 0 |
| INDAN | 0 | NS | NS | NS | NS | 0 |
| UNDECANE | 72 | 72 | 80 | 76 | 85 | 75 |
| NAPHTHALENE | 50 | 54 | 39 | 0 | 57 | 37 |
| DODECANE | 83 | 85 | 92 | 90 | 97 | 88 |
| TRIDECANE | 89 | 87 | 93 | 93 | 98 | 91 |
| TETRADECANE | 100 | 100 | 100 | 100 | 100 | 100 |
| PENTADECANE | 114 | 104 | 103 | NS | 140 | 125 |
| HEXADECANE | NS | NS | NS | NS | NS | 0 |

[a] NS means insufficient sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was available to ascertain the presence or absence of the hydrocarbon. Detection limits were different for each hydrocarbon because of the relative sensitivity of the gas chromatograph detector response.

[b] Sample size (based on the concentration of tetradecane, the persistent hydrocarbon used as the internal standard) was large enough, but hydrocarbon concentration below detection limits. Detection limits were different for each hydrocarbon.



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# APPENDIX C

# DATA TABLES (MISSILE FUEL STUDY)

TABLE C-1. MEASURED CONCENTRATIONS OF HYDROCARBONS IN JP-9 ACTIVE WATER FLASKS: RANGE POINT

| Compound[b] | CONCENTRATIONS (MG/L)[a] Time (Hours) 0 | 2 | 4 | 8 | 24 | 60 | 86 | 120 |
|---|---|---|---|---|---|---|---|---|
| HNN | 56.23 ±3.25 | 58.06 ±8.87 | 58.86 ±3.58 | 58.88 ±6.40 | 49.58 ±5.98 | 6.01 ±0.77 | 8.22 ±7.94 | 0.18 ±0.42 |
| XTHDCPD | 167.07 ±8.21 | 85.86 ±23.45 | 79.26 ±22.22 | 15.35 ±7.91 | 1.27 ±1.43 | ND | ND | ND |
| NTHDCPD | 3.81 ±0.13 | 2.08 ±0.59 | 2.02 ±0.54 | 0.33 ±0.32 | ND | ND | ND | ND |
| 2,5-Dimethylhexane | 1.06 ±0.11 | ND[c] | ND | ND | ND | ND | ND | ND |
| Toluene | 1.46 ±0.11 | ND | ND | ND | ND | ND | ND | ND |
| Heptane | 1.46 ±0.08 | ND | ND | ND | ND | ND | ND | ND |
| Methylcyclohexane | 11.07 ±0.87 | 0.43 ±0.01 | 0.22 ±0.44 | ND | ND | ND | ND | ND |

[a] Concentrations are the means of duplicate samples and standard deviation; stars where shown indicate only single samples and therefore standard deviation could not be calculated.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); XTHDCPD, exo-tetrahydrodi (cyclopentadiene); NTHDCPD, endo-tetrahydrodi (cyclopentadiene).

[c] ND = < 0.1 mg/L

TABLE C-2. MEASURED CONCENTRATIONS OF HYDROCARBONS IN JP-9 STERILE WATER FLASKS: RANGE POINT

| Compound[b] | Time (Hours) | CONCENTRATIONS (mg/L)[a] 0 | 2 | 4 | 8 | 24 | 60 | 86 | 120 |
|---|---|---|---|---|---|---|---|---|---|
| HNN | | 0.29 ±68.46 | 56.99 * | 56.21 ±3.24 | 62.34 ±2.79 | 45.94 ±0.20 | 14.53 ±0.00 | 12.78 ±11.01 | 0.23 ±0.00 |
| XTHDCPD | | 211.23 ±2.84 | 84.81 * | 89.28 ±1.99 | 27.05 ±5.85 | 2.12 ±2.77 | ND | ND | ND |
| NTHDCPD | | 4.16 ±1.15 | 1.69 * | 2.12 ±0.02 | 0.53 ±0.01 | ND | ND | ND | ND |
| 2,5-Dimethylhexane | | 1.35 ±0.12 | 0.22 * | 0.20 ±0.01 | ND | ND | ND | ND | ND |
| Toluene | | 1.60 ±0.04 | 0.25 * | 0.27 ±0.03 | ND | ND | ND | ND | ND |
| Heptane | | 1.64 ±0.02 | ND[c] | ND | ND | ND | ND | ND | ND |
| Methylcyclohexane | | 11.98 ±0.10 | 0.50 * | 0.30 ±0.00 | ND | ND | ND | ND | ND |

[a] Concentrations are the means of duplicate samples and standard deviation; stars where shown indicate only single samples and therefore standard deviation could not be calculated.

[b] HNN, endo, endo-tetrahydrodi-norbornadiene); XTHDCPD, exo-tetrahydrodi (cyclopentadiene); NTHDCPD, endo-tetrahydrodi (cyclopentadiene).

[c] ND = < 0.1 mg/l

TABLE C-3. MEASURED CONCENTRATIONS OF HYDROCARBONS IN JP-9 IN ACTIVE SEDIMENT FLASKS: RANGE POINT

| Compound[b] | Time (Hours) | CONCENTRATIONS (mg/L)[a] 0 | 2 | 4 | 8 | 24 | 60 | 86 | 120 |
|---|---|---|---|---|---|---|---|---|---|
| HNN | | 58.97 ±4.68 | 53.11 ±0.13 | 59.58 * | 47.98 ±0.47 | 40.29 * | 26.25 ±4.33 | 25.34 ±0.93 | 19.49 ±2.39 |
| XTHDCPD | | 171.21 ±19.38 | 102.60 ±5.90 | 112.70 * | 86.02 ±5.19 | 86.55 * | 58.78 ±11.13 | 56.94 ±3.83 | 41.36 ±5.74 |
| NTHDCPD | | 3.81 ±0.47 | 2.28 ±0.10 | 2.53 * | 1.85 ±0.12 | 1.89 * | 1.22 ±0.22 | 1.04 ±0.11 | 0.85 ±0.12 |
| 2,5-Dimethylhexane | | 1.30 ±0.17 | 0.16 ±0.04 | 0.21 * | 0.15 ±0.00 | 1.15 * | ND | ND | ND |
| Toluene | | 1.45 ±0.13 | 0.19 ±0.02 | 0.29 * | ND[c] | ND | ND | ND | ND |
| Heptane | | 1.62 ±0.13 | 0.13 ±0.03 | 0.17 * | 0.14 ±0.00 | 0.17 * | ND | ND | ND |
| Methylcyclohexane | | 12.07 ±1.35 | 1.47 ±0.13 | 1.95 * | 1.16 ±0.16 | 1.14 * | 0.15 ±0.01 | 0.23 ±0.19 | 0.15 ±0.05 |

[a] Concentrations are the means of duplicate samples and standard deviation; stars where shown indicate only single samples and therefore standard deviation could not be calculated.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); XTHDCPD, exo-tetrahydrodi (cyclopentadiene); NTHDCPD, endo-tetrahydrodi (cyclopentadiene).

[c] ND = < 0.1 mg/L

TABLE C-4. MEASURED CONCENTRATIONS OF HYDROCARBONS IN JP-9 STERILE SEDIMENT FLASKS: RANGE POINT

| Compound[b] | CONCENTRATIONS (mg/L)[a] Time (Hours) 0 | 2 | 4 | 8 | 24 | 60 | 86 | 120 |
|---|---|---|---|---|---|---|---|---|
| HNN | 53.34 ±2.83 | 51.16 ±0.34 | 58.62 * | 42.37 ±10.55 | 40.14 * | 25.30 ±0.01 | 22.68 ±2.21 | 24.72 ±0.88 |
| XTHDCPD | 156.54 ±17.33 | 116.03 ±0.45 | 128.58 * | 109.83 ±5.81 | 71.04 * | 58.11 ±2.59 | 49.08 ±4.67 | 54.45 ±1.80 |
| NTHDCPD | 2.55 ±0.22 | 1.86 ±0.08 | 1.96 * | 1.81 ±0.43 | 1.07 * | 0.79 ±0.01 | 1.19 ±0.31 | 0.77 ±0.05 |
| 2,5-Dimethylhexane | 1.47 ±0.18 | 0.21 ±0.01 | 0.26 ±0.02 | 0.21 | 0.16 | ND | ND | ND |
| Toluene | 1.36 ±0.33 | 0.31 ±0.15 | 0.31 * | ND[c] | ND | ND | ND | ND |
| Heptane | 1.44 ±0.21 | 0.16 ±0.00 | 0.18 * | 0.16 ±0.03 | ND | ND | ND | ND |
| Methylcyclohexane | 10.75 ±1.71 | 1.63 ±0.22 | 2.03 * | 1.56 ±0.16 | 1.03 * | 0.35 ±0.06 | 0.22 ±0.03 | 0.21 ±0.03 |

[a] Concentrations are the means of duplicate samples and standard deviation; stars where shown indicate only single samples and therefore standard deviation could not be calculated.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); XTHDCPD, exo-tetrahydrodi (cyclopentadiene); NTHDCPD, endo-tetrahydrodi (cyclopentadiene).

[c] ND = < 0.1 mg/L

TABLE C-5. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 ACTIVE WATER FLASKS: ESCAMBIA RIVER

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 480 | 672 | 816 | 963 | 1128 | 1368 |
|---|---|---|---|---|---|---|---|---|---|
| HNN | | 369.06 ±20.42 | 367.12 ±30.99 | 369.29 ±53.92 | 278.65 ±36.07 | 240.28 ±46.57 | 298.73 ±41.58 | NS[c] | 162.74 ±126.13 |
| HXN | | 7.80 ±0.49 | 7.66 ±0.77 | 7.86 ±1.34 | 6.14 ±0.73 | 5.03 ±1.01 | 6.36 ±0.95 | NS | 3.42 ±2.69 |
| DHNN | | 4.30 ±0.33 | 4.09 ±0.50 | 4.33 ±0.76 | 3.27 ±0.42 | 2.78 ±0.59 | 3.47 ±0.53 | NS | 1.80 ±1.50 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

[c] NS; no sample

TABLE C-6. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 STERILE WATER FLASKS: ESCAMBIA RIVER

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 480 | 672 | 816 | 963 | 1128 | 1368 |
|---|---|---|---|---|---|---|---|---|---|
| HNN | | 386.30 ±16.04 | 374.83 ±25.57 | 388.16 ±49.83 | 338.56 ±75.52 | 339.78 ±19.85 | 408.50 ±25.91 | 352.97 ±20.74 | 323.27 ±12.22 |
| HXN | | 7.99 ±0.50 | 7.74 ±0.65 | 8.00 ±1.08 | 7.15 ±1.36 | 7.10 ±0.43 | 7.92 ±0.36 | 7.56 ±0.51 | 6.63 ±0.30 |
| DHNN | | 4.46 ±0.32 | 4.26 ±0.46 | 4.53 ±0.68 | 3.96 ±0.78 | 3.88 ±0.24 | 4.35 ±0.18 | 4.04 ±0.32 | 3.66 ±0.17 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

TABLE C-7. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 ACTIVE SEDIMENT: ESCAMBIA RIVER

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 480 | 672 | 816 | 963 | 1128 | 1368 |
|---|---|---|---|---|---|---|---|---|---|
| HNN | | 351.46 ±30.93 | 302.09 ±82.70 | 234.42 ±101.40 | 318.71 ±9.29 | 132.25 ±57.29 | 79.16 ±28.20 | 193.45 ±136.54 | 108.18 ±7.14 |
| HXN | | 7.45 ±0.69 | 6.52 ±1.72 | 4.80 ±2.29 | 5.66 ±1.42 | 2.77 ±1.20 | 4.68 ±1.65 | 4.13 ±0.84 | 2.23 ±0.17 |
| DHNN | | 4.06 ±0.43 | 3.28 ±0.93 | 2.60 ±1.26 | 3.82 ±0.11 | 1.52 ±0.70 | 0.90 ±0.34 | 2.26 ±0.42 | 1.17 ±0.07 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

TABLE C-8. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 STERILE SEDIMENT FLASKS: ESCAMBIA RIVER

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 480 | 672 | 816 | 963 | 1128 | 1368 |
|---|---|---|---|---|---|---|---|---|---|
| HNN | | 377.19 ±27.49 | 379.04 ±24.13 | 166.37 ±90.02 | 150.02 ±20.10 | 243.00 ±10.09 | 235.85 ±25.88 | 189.98 ±28.00 | 208.87 ±89.86 |
| HXN | | 7.92 ±0.63 | 7.84 ±0.61 | 3.11 ±1.51 | 4.21 ±2.48 | 5.12 ±0.30 | 4.49 ±0.37 | 4.08 ±0.62 | 4.33 ±1.92 |
| DHNN | | 4.43 ±0.43 | 4.30 ±0.36 | 2.39 ±0.13 | 2.30 ±1.39 | 2.82 ±0.17 | 2.45 ±0.22 | 2.23 ±0.34 | 2.39 ±1.06 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

TABLE C-9. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 ACTIVE WATER FLASKS: RANGE POINT

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 | 22 | 72 | 144 | 192 | 288 | 521 | 689 | 984 | 1320 |
| HNN | | 380.91 ±13.82 | 475.24 ±6.85 | 345.06 ±26.07 | 328.02 ±18.92 | 307.65 ±5.97 | 381.97 ±15.59 | 340.58 ±26.29 | 324.71 ±88.17 | 343.89 ±25.63 | 353.98 ±7.41 |
| HXN | | 8.02 ±0.23 | 9.90 ±0.11 | 7.17 ±0.67 | 6.59 ±0.11 | 6.43 ±0.16 | 8.00 ±0.35 | 6.98 ±0.22 | 6.89 ±1.86 | 7.23 ±0.56 | 7.38 ±0.19 |
| DHNN | | 4.48 ±0.21 | 5.54 ±0.10 | 4.01 ±0.43 | 3.35 ±0.17 | 3.51 ±0.09 | 4.49 ±0.18 | 3.58 ±0.20 | 3.91 ±1.11 | 3.99 ±0.37 | 4.09 ±0.12 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

TABLE C-10. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 STERILE WATER FLASKS: RANGE POINT

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 72 | 144 | 192 | 288 | 521 | 689 | 984 | 1320 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| HNN | | 396.22 ±11.71 | 375.37 ±5.10 | 353.37 ±22.38 | 357.01 ±2.83 | 296.27 ±8.34 | 306.44 ±74.91 | 353.89 ±44.88 | 373.29 ±33.86 | 336.95 ±14.50 | 353.76 ±5.58 |
| HXN | | 8.35 ±0.25 | 7.88 ±0.04 | 7.29 ±0.50 | 7.43 ±0.13 | 6.20 ±0.15 | 6.48 ±1.50 | 6.96 ±0.96 | 7.66 ±0.70 | 7.06 ±0.28 | 7.42 ±0.12 |
| DHNN | | 4.64 ±0.22 | 4.49 ±0.06 | 4.01 ±0.27 | 4.06 ±0.13 | 3.38 ±0.08 | 3.60 ±0.83 | 3.78 ±0.58 | 4.28 ±0.33 | 3.69 ±0.16 | 4.16 ±0.14 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

TABLE C-11. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 ACTIVE SEDIMENT FLASKS: RANGE POINT

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 72 | 144 | 192 | 288 | 521 | 689 | 984 | 1320 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| HNN | | 491.57 ±131.43 | 449.02 ±42.96 | 357.80 ±8.72 | 349.36 ±11.47 | 267.46 ±6.22 | 344.25 ±11.08 | 352.52 ±22.69 | 366.59 ±18.61 | 405.49 ±20.68 | 328.38 ±15.18 |
| HXN | | 10.30 ±2.75 | 9.37 ±0.87 | 7.58 ±0.21 | 7.34 ±0.37 | 5.60 ±0.12 | 7.34 ±0.19 | 7.44 ±0.52 | 7.76 ±0.40 | 8.47 ±0.48 | 6.70 ±0.21 |
| DHNN | | 5.41 ±1.35 | 5.24 ±0.57 | 4.23 ±0.11 | 3.99 ±0.32 | 3.06 ±0.06 | 3.99 ±0.08 | 3.69 ±0.30 | 4.35 ±0.21 | 4.74 ±0.29 | 3.73 ±0.12 |

[a] Concentrations are the means of duplicate samples and standard deviation.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

TABLE C-12. MEASURED CONCENTRATIONS OF HYDROCARBONS IN RJ-5 STERILE SEDIMENT FLASKS: RANGE POINT

| Compound[b] | Time (Hours) | CONCENTRATION (ug/L)[a] 0 | 22 | 72 | 144 | 192 | 288 | 521 | 689 | 984 | 1320 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| HNN | | 391.46 ±7.85 | 364.15 ±18.31 | 368.10 ±16.42 | 364.37 ±12.76 | 269.39 ±43.10 | 359.60 ±8.80 | 364.01 * | 370.89 ±63.03 | NS[c] | NS |
| HXN | | 8.35 ±0.23 | 7.56 ±0.39 | 7.77 ±0.38 | 7.52 ±0.28 | 5.51 ±0.52 | 7.66 ±0.11 | 7.69 * | 7.70 ±1.31 | NS | NS |
| DHNN | | 4.63 ±0.22 | 4.09 ±0.33 | 4.37 ±0.25 | 4.04 ±0.16 | 3.04 ±0.53 | 4.24 ±0.09 | 4.33 * | 4.02 ±0.54 | NS | NS |

[a] Concentrations are the means of duplicate samples and standard deviation; stars indicate only single samples and therefore a standard deviation could not be calculated.

[b] HNN, endo, endo-dihydrodi-(norbornadiene); HXN, exo, endo-dihydrodi-(norbornadiene); DHNN, dehydro-endo, endo-dihydrodi-(norbornadiene).

[c] NS; no sample

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